U.S. DEPARTMENT OF COMMERCE
DANIEL C. ROPER, Secretary
BUREAU OF STANDARDS
LYMAN J. BRIGGS, Director

BUREAU OF STANDARDS JOURNAL OF RESEARCH

October 1933

Vol. 11, No. 4

UNITED STATES
GOVERNMENT PRINTING OFFICE
WASHINGTON : 1933

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U.S. Department of Commerce, Bureau of Standards

RESEARCH PAPER RP600

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

EFFECT OF WEAVE ON THE PROPERTIES OF CLOTH[1]

By Herbert F. Schiefer, Richard S. Cleveland, John W. Porter, and Joshua Miller

ABSTRACT

The effect of the weave on the strength, elongation, take-up, tear resistance, fabric assistance, and air permeability of cloth is discussed in this paper. For this purpose a series of 42 cloths were woven from the same cotton yarns in weaves comprising plain, twill, rib, mock leno, basket, sateen, and various combinations of these weaves. The factors which contribute to strength and tear resistance are enumerated and discussed.

Four cloths having high tear resistance were woven for experiments on rubberizing and on doping. The results of these experiments are given and compared with the properties of the gas cell cloth and outer cover cloth which are used in dirigible construction.

CONTENTS

	Page
I. Introduction	441
II. Description of cloths	442
III. Measurement of physical properties	442
1. Thread count	445
2. Weight	445
3. Breaking strength and elongation	445
4. Take-up	445
5. Tear resistance	446
6. Air permeability	446
IV. Discussion	446
V. Application	450
VI. Summary	451

I. INTRODUCTION

The physical properties and the appearance of cloth depend to a great extent upon the weave. The strength, elongation, take-up, tear resistance, fabric assistance, air permeability, and texture are some of the properties which may be varied by changing the weave. For aeronautical cloths with rigid limits on the weight, the weave is an important means of obtaining some of the desired physical properties.

A cloth of plain weave has in general a greater breaking strength and a lower tear resistance than cloths of the same weight but of different weaves. It also has a greater take-up, higher elongation, and the shortest floats.[2] Not only are high breaking strength and short floats essential in aeronautical cloths, but also high tear resistance and in some cases low elongation.

The purpose of this investigation was to weave a series of cloths from the same cotton yarns in several weaves and to determine the

[1] This investigation was started by Mr. Miller before he left the Bureau of Standards. The cloths were woven by Mr. Porter.

[2] "Float" means the length of a warp or filling yarn between successive interlacings.

effect on the physical properties. A specific object was the determination of the factors requisite for high tear resistance. Four cloths of high tear resistance were woven for experiments on rubberizing and on doping. The results are given and compared with the properties of the untreated cloths and with the properties of the gas cell cloth and outer cover cloth used in dirigible construction.

This work was undertaken by the Bureau of Standards at the request of and with the financial assistance of the Bureau of Aeronautics, Department of Navy. The experiments on rubberizing and doping were carried out by the Goodyear Zeppelin Corporation. Their assistance and cooperation are gratefully acknowledged.

II. DESCRIPTION OF CLOTHS

The cloths were woven on one loom and with approximately the same tension. The number of threads per inch of cloth in the loom was 90 in the warp and in the filling. The yarn used in the warp was a 57's cotton having a twist of 31 turns per inch (twist multiplier 4). The yarn used in the filling was a 60's cotton having a twist of 21 turns per inch (twist multiplier 2.6). The yarns were of ordinary commercial quality and were bought in the open market.

The weave designs of the cloths, comprising plain, twill, rib, mock leno, basket, sateen, and combinations of these weaves are shown in figures 1, 2, and 3. In drawing the designs the customary convention was employed, namely, a black square indicates that the warp yarn is raised and appears on the upper surface, while a white square indicates that the filling yarn is raised and appears on the upper surface. The vertical columns represent the warp direction while the horizontal rows represents the filling direction. At least one complete pattern of each design is given.

Photomicrographs of the cloths are also shown in figures 1, 2, and 3. They exhibit the surface characteristics of the cloths.

The weaves corresponding to the numbers in figures 1, 2, and 3 are given in table 1. The texture and outstanding surface characteristics are also noted. The cloth of each weave is classified either as firm or sleazy. The cloth is said to be firm if the warp yarns are not easily distorted relative to the filling yarns and sleazy if they are easily distorted. The cloth of each weave is classified either as close or open. The cloth is said to be close if the interstices formed by the interlacing of warp and filling yarns are small and open if they are large. The cloth of each weave is classified either as having long floats or short floats. The cloth is said to have long warp floats if the sum of the number of consecutive filling yarns which a warp yarn passes over plus the number of consecutive filling yarns which it passes under is equal to or greater than 8. A similar convention is used for determining long filling floats. These are purely arbitrary conventions adopted for the purpose of classification.

III. MEASUREMENT OF PHYSICAL PROPERTIES

All tests were made on material having a normal moisture content, obtained by proper exposure to atmospheric conditions of 65 percent relative humidity and 70° F. Unless otherwise stated, 10 determinations were made for each test. The average result for each test is given in table 1 for the various cloths.

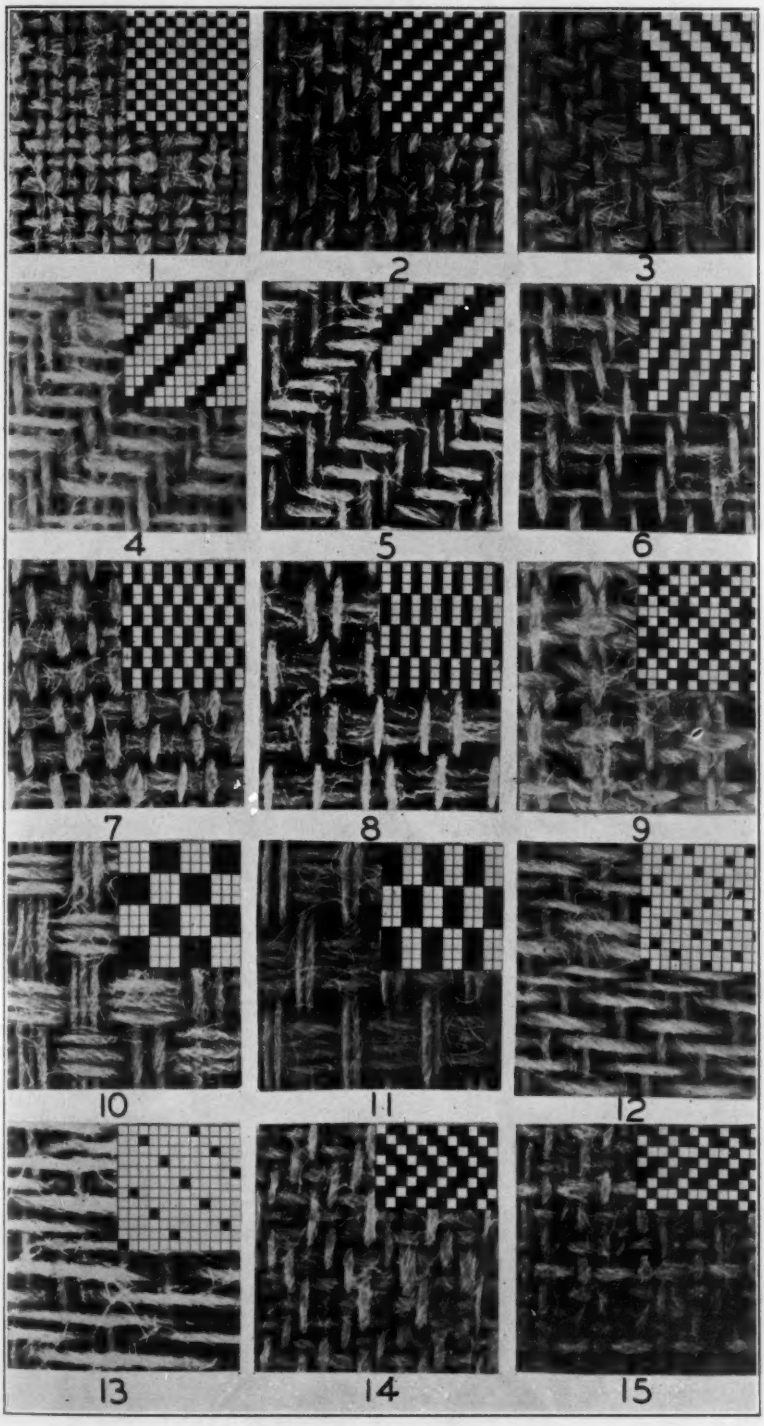

FIGURE 1.—*Weave designs and photomicrographs* ($\times 11$) *of the cloths.*

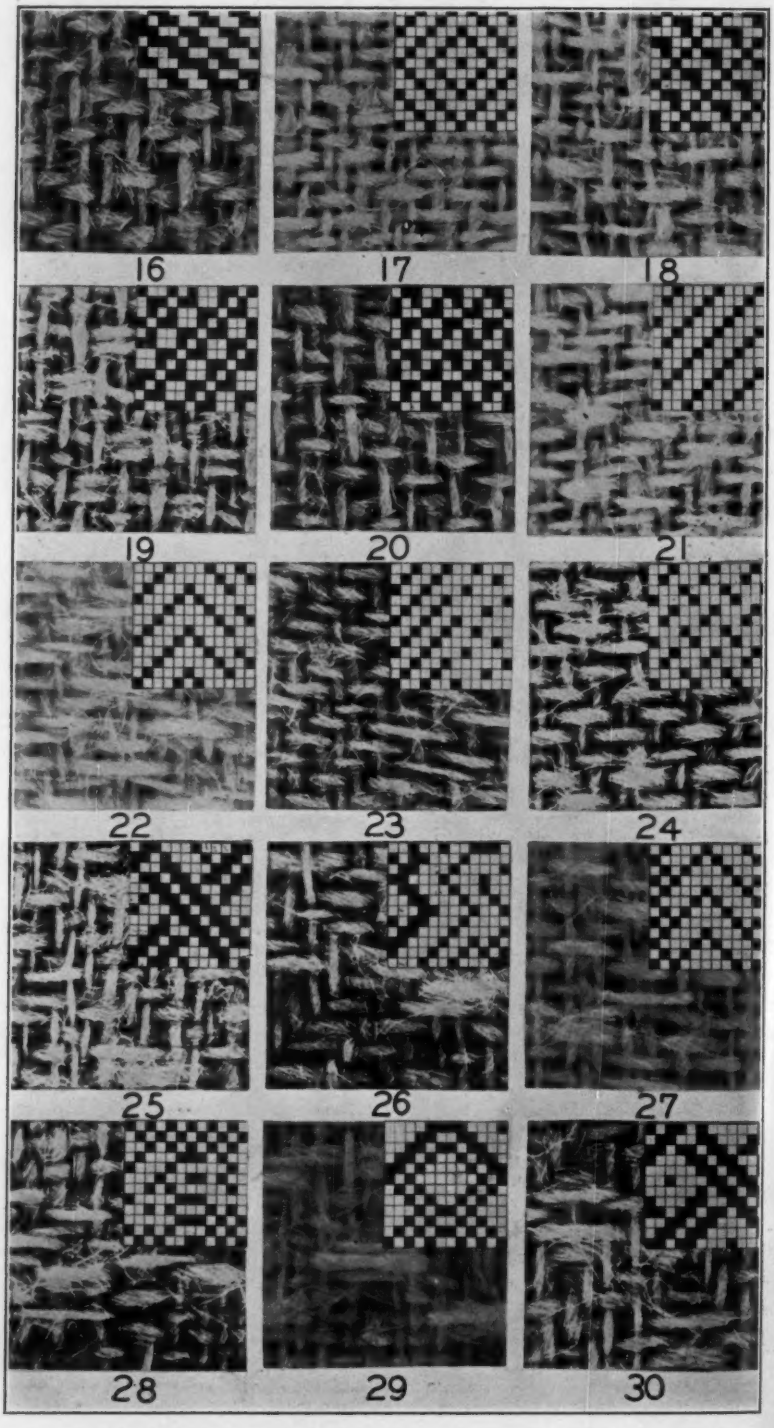

FIGURE 2.—*Weave designs and photomicrographs* ($\times 11$) *of the cloths.*

TABLE 1.—*Effect of weave on the properties of cloths*

Type of weave	Texture and surface characteristics						Specimen no.	Threads per inch		Weight per square yard [1]	Breaking strength [1]		Fabric assistance		Elongation at rupture		Take-up		Tear resistance		Air permeability
	Firm, F	Sleazy, S	Close, C	Open, O	Short floats, s	Long floats, L		Warp	Filling		Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	
										Oz.	*Lb.* [2] 35	*Lb.* [2] 16	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Lb.*	*Lb.*	
Yarn																					
Plain	F		C		s		1	96	94	2.28	40	33	14	106	11	12	8	8	2.5	2	[3] 192
Twill	F		C		s		2	96	92	2.23	38	26	9	63	9	12	6	8	3.5	2	245
Do	F		C		s		3	96	91	2.22	33	23	−6	44	7	11	3	7	4	2	307
Do		S		O	s		4	96	89	2.26	32	20	−9	25	5	10	3	7	4.5	2.5	405
Do		S		O	s		5	96	90	2.22	35	21	0	31	5	11	2	7	4.5	2.5	400
Do		S		O	s		6	93	92	2.21	42	22	20	38	9	8	5	3	4.5	3	405
Rib	F			O	s		7	92	93	2.19	42	25	20	56	14	7	8	2	3.5	3	362
Do		S		O	s		8	92	92	2.22	34	22	−3	38	10	7	6	2	4.5	3.5	451
Mock Leno	F			O	s		9	95	92	2.29	35	25	0	56	9	11	6	6	4.5	3	431
Basket		S		O	s		10	94	91	2.18	29	19	−17	19	5	8	2	6	5.5	3	383
Do		S		O		L	11	93	91	2.17	35	22	0	38	7	7	3	3	5	3.5	451
Sateen		S	C		s		12	95	90	2.22	39	24	11	50	6	10	3	5	4.5	3	455
Do		S		O		L	13	93	90	2.21	37	22	6	38	5	8	2	5	6	4	633
Combination	F		C		s		14	95	93	2.25	38	27	9	69	11	11	6	7	3.5	2.5	261
Do	F		C		s		15	96	93	2.24	40	30	14	87	10	12	6	7	3.5	2.5	217
Do	F		C		s		16	96	92	2.23	40	29	14	81	8	13	4	7	4	2.5	285
Do	F		C		s		17	95	92	2.22	40	28	14	75	9	10	5	7	3.5	3.5	283
Do	F		C		s		18	95	93	2.23	39	26	11	63	10	11	5	6	4	2.5	313
Do	F		C		s		19	95	93	2.23	40	26	14	63	10	11	5	6	3.5	2.5	266
Do	F		C		s		20	96	92	2.24	40	28	14	75	9	12	5	8	4	2.5	272
Do	F		C		s		21	96	91	2.23	39	27	11	69	8	11	4	6	4.5	2.5	325
Do	F		C		s		22	95	92	2.22	38	25	9	56	8	10	4	5	4	2.5	319
Do	F		C		s		23	96	91	2.23	39	26	11	63	7	11	3	6	4	2.5	336
Do	F		C		s		24	96	91	2.26	39	26	11	63	7	11	4	6	4.5	2.5	325
Do	F		C		s		25	95	93	2.23	39	26	11	63	10	11	5	6	4	2.5	307
Do	F		C			L	26	96	92	2.23	39	27	11	69	7	11	4	7	4	2.5	378

[1] Corrected to 90 by 90 thread count basis.
[2] Breaking strength of 90 threads by the multiple strand method.
[3] Cubic feet per minute per square foot of cloth at a pressure of 0.5 inch of water across the cloth.

TABLE 1.—*Effect of weave on the properties of cloths*—Continued

Type of weave	Texture and surface characteristics						Specimen no.	Threads per inch		Weight per square yard	Breaking strength [1]		Fabric assistance		Elongation of rupture		Take-up		Tear resistance		Air permeability
	Firm, F	Sleazy, S	Close, C	Open, O	Short floats, s	Long floats, L		Warp	Filling		Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling	
										Oz.	*Lb.*	*Lb.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Lb.*	*Lb.*	
Do	F		C		s		27	96	92	2.23	40	29	14	81	9	11	5	7	4	2.5	290
Do	F		C			L	28	96	92	2.22	40	28	14	75	9	10	4	5	4	2.5	331
Do	F		C			L	29	95	91	2.23	39	27	11	69	8	12	4	6	4	2.5	341
Do	F		C		s		30	96	91	2.19	33	22	−6	38	7	10	4	7	4	2.5	336
Do	F			O	s		31	95	91	2.22	33	24	−6	50	7	10	4	7	4.5	2.5	373
Do	F		C		s		32	96	90	2.27	32	25	−9	56	7	10	3	6	4.5	2.5	313
Do	F		C		s		33	95	92	2.22	34	23	−3	44	7	10	3	5	4.5	2.5	373
Do	F			O	s		34	96	92	2.19	34	22	−3	38	6	10	3	7	4.5	2.5	357
Do		S		O	s		35	94	92	2.20	40	26	14	63	9	9	4	4	4.5	3	396
Do		S		O	s		36	96	90	2.28	39	26	11	63	7	11	3	7	5	3	550
Do		S		O		L	37	94	92	2.23	30	22	−14	38	6	10	3	6	4.5	3	414
Do		S	C			L	38	95	93	2.21	39	26	11	63	8	10	4	7	4	2.5	367
Do		S	C			L	39	95	90	2.25	32	22	−9	38	7	10	3	6	4.5	2.5	435
Do		S		O		L	40	95	91	2.20	38	26	9	63	6	10	2	6	5.5	3	387
Do		S		O		L	41	93	92	2.16	28	20	−20	25	6	9	3	4	5	3	514
Do		S		O		L	42	92	91	2.15	33	18	−6	13	5	7	2	2	5.5	3.5	489

[1] Corrected to 90 by 90 thread count basis.
[2] Breaking strength of 90 threads by the multiple strand method.
[3] Cubic feet per minute per square foot of cloth at a pressure of 0.5 inch of water across the cloth.

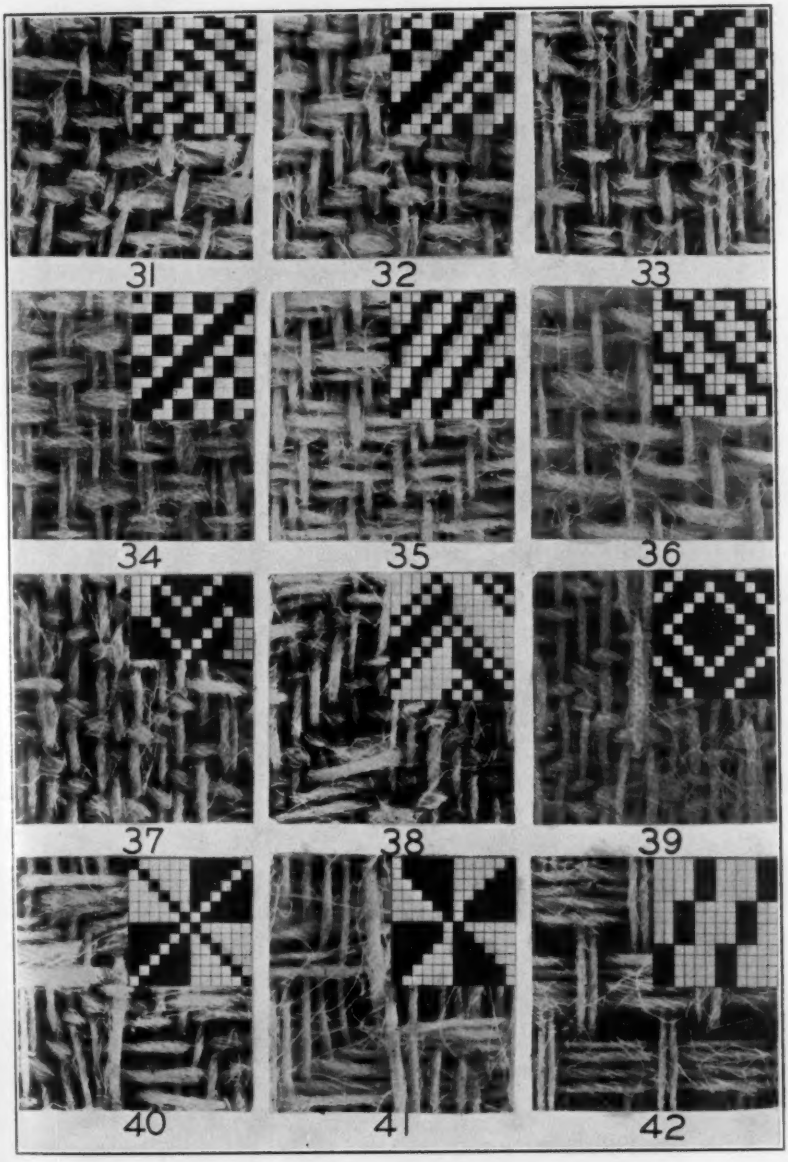

FIGURE 3.—*Weave designs and photomicrographs* (× *11*) *of the cloths.*

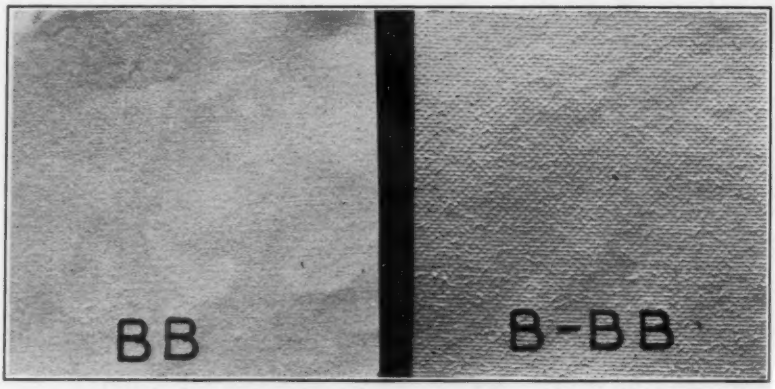

FIGURE 8.—*Surface characteristics of doped cloths.*

BB is regular doped outer cover cloth for dirigibles; B-BB is Bureau of Standards high tear resistance doped cloth.

1. THREAD COUNT

Thread-count determinations were made on the specimens which were used for breaking strength. Because of the contraction of the cloth when the tension on it in the loom was released, the number of threads per inch in both the warp and filling is greater than 90. The variation from 90 depends upon the weave and is a maximum for the plain weave.

2. WEIGHT

In weight determinations specimens 2 inches square were cut with a die and weighed on an analytical balance. The weight was then expressed in ounces per square yard. The values reported in table 1 have been reduced for comparison to a thread-count basis of 90 threads per inch in warp and filling, respectively.[3] The weights vary from 2.15 to 2.29 ounces per square yard. The variation is due to the different degree of waviness produced in the yarns by the various weaves. The degrees of waviness of the yarns in the various weaves may be seen in figures 1, 2, and 3.

3. BREAKING STRENGTH AND ELONGATION

Rectangular specimens, approximately 6 by 1¼ inches, were cut with the long edge either in the warp or in the filling direction as desired. The specimens were then frayed to 1 inch in width and the threads were counted (see section 1).

The tests were made according to the standard procedure on an inclination balance type of machine equipped with an autographic device for drawing the stress-strain curve. The breaking strengths are reported in table 1. For comparison they have been reduced to a thread-count basis of 90 threads per inch. The strengths of 90 warp and of 90 filling yarns tested by the multiple-strand method are also given.[4] The difference between the breaking strength of the cloth on the basis of 90 threads per inch and the breaking strength of 90 strands of yarn expressed as a percentage of the breaking strength of 90 strands of yarn, known as fabric assistance, is also reported in table 1.

The elongation at the breaking load was read directly on the chart from the stress-strain curve. The elongation at rupture in percent is reported in table 1.

4. TAKE-UP

Take-up, that is, the percentage decrease of yarn length in the cloth because of the waviness resulting from yarn interlacing, was determined by making ink marks on the cloth 10 inches apart along the warp and filling, respectively. The yarn was then removed from the cloth and the length between the ink marks was measured when just sufficient tension was applied to remove the waviness. If L represents this length in inches, then take-up is given by the expression $(L-10)\frac{100}{L}$. The results are given in table 1.

[3] In applying the correction it was assumed that the warp and filling yarns had the same weight.

[4] C. W. Schoffstall and H. A. Hamm, A Multiple Strand Test for Yarns. Bureau of Standards Journal of Research, vol. 2, no. 5, pp. 871-885, 1929.

5. TEAR RESISTANCE

Tear resistance determinations were made on specimens 3 inches wide and 6 inches long according to the preferred method of the American Society for Testing Materials.[5] The average result of 5 tests in the warp direction and of 5 tests in the filling direction is reported in table 1 for each cloth.

6. AIR PERMEABILITY

Air-permeability determinations were made according to the method described by Schiefer and Best.[6] The average result of 3 tests for each cloth is reported in table 1. It is expressed in cubic feet per

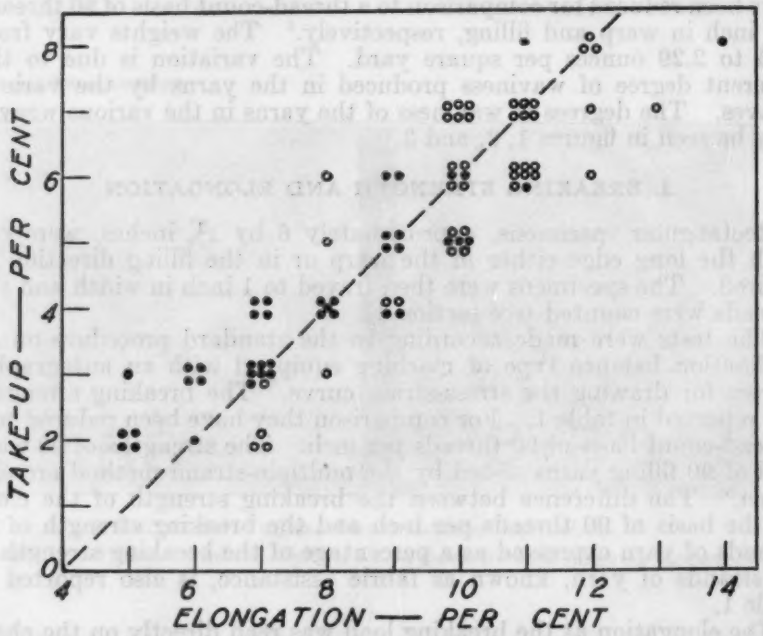

FIGURE 4.—*Correlation between elongation and take-up.*

The points plotted represent the elongation and take-up of the cloths. The values for the warp are indicated by dots and for the filling by circles.

minute per square foot of cloth at a pressure drop of 0.5 inch of water across the cloth.

IV. DISCUSSION

The data in table 1 show that the weave has a considerable effect upon the properties of the cloths. It is difficult to correlate the properties with the type of weave since the variations of weave for a given type, twill for example, produce almost as great variations as between the types. However, it is worth while to call attention to the correlations between some of the properties.

[5] This is the alternate method described in Proceedings of the American Society for Testing Materials, vol. 32, part I, p. 1007, 1932. It was changed to the preferred method at their annual meeting in Chicago, June 26, 1933.

[6] H. F. Schiefer and A. S. Best, A Portable Instrument for Measuring Air Permeability of Fabrics, B.S. Jour. Research, vol. 6 (RP261), pp. 51–58, 1931.

The elongation of the cloth at rupture, which varies from 5 to 14 percent, appears to vary directly with the take-up (see fig. 4). In general the elongation is about 4 percent greater than the take-up. This increase may be ascribed to the elongation of the yarn.

TABLE 2.—*Effect of texture and surface characteristics on the properties of cloth* [a]

Texture and surface characteristics	Breaking strength		Fabric assistance		Elongation at rupture		Take-up		Tear resistance		Air permeability
	W	F	W	F	W	F	W	F	W	F	
	Lb.	*Lb.*	%	%	%	%			*Lb.*	*Lb.*	
Firm	37.8	26.3	8	64	8.6	10.7	4.6	6.3	3.9	2.5	[b] 312
Sleazy	35.1	22.4	0	40	6.6	9.0	3.1	5.0	4.8	3.0	445
Close	38.3	26.8	9	68	8.7	11.1	4.7	6.6	3.9	2.5	292
Open	35.4	22.9	1	43	7.1	9.3	3.5	5.2	4.7	2.9	428
Short floats	37.3	25.2	7	57	8.3	10.4	4.4	6.1	4.1	2.6	339
Long floats	35.4	23.6	1	48	6.7	9.5	3.1	5.2	4.8	3.0	431
Firm, close, short floats	38.2	26.8	9	68	8.7	11.1	4.7	6.7	3.9	2.5	287
Sleazy, open, long floats	34.0	22.2	−3	39	6.3	8.9	2.8	3.6	5.0	3.1	462

[a] The data of this table are obtained by taking the average of the values reported in table 1 for each classification of texture and surface characteristics.

[b] Cubic feet per minute per square foot of cloth at a pressure drop of 0.5 inch of water across the cloth.

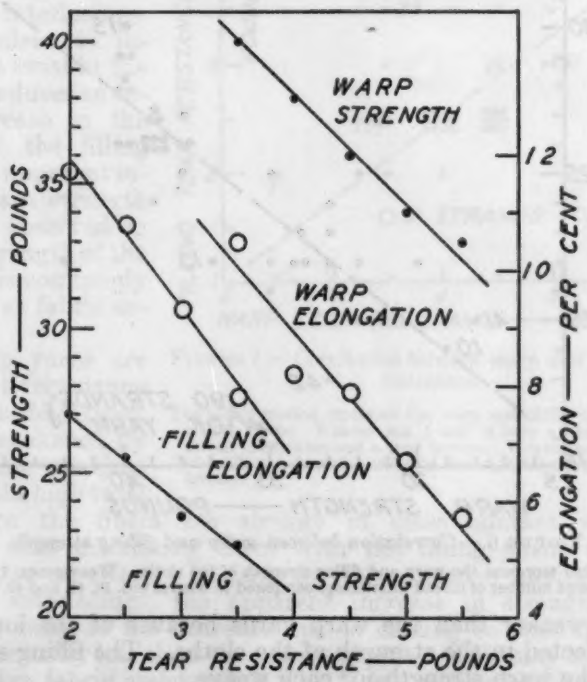

FIGURE 5.—*Correlations of strength and elongation with tear resistance.*

The points plotted represent the average strength and the average elongation of cloths having the same tear resistance.

The data in table 2 show the effect of a firm or sleazy texture, of a close or open weave, and of short or long floats upon the properties of the cloth. In general a cloth which is firm, close, and has short floats has greater strength, fabric assistance, elongation, and take-up and has smaller tear resistance and air permeability than one which is sleazy, open and has long floats.

In general the strength and elongation decrease as the tear resistance is increased. The decrease in average strength and average elongation with increase in tear resistance is shown in figure 5.

It is worth while to discuss briefly some of the factors which contribute to the strength, fabric assistance, and tear resistance of a cloth.

The factors which contribute to the strength of a cloth are (1) the strength of the yarn, (2) the manner of the interlacing of the one set of yarns relative to the other set of yarns, and (3) the twist of the yarn.

It is well known that the strength of a cotton yarn increases with twist up to a certain optimum twist, namely, that which is necessary to practically eliminate fiber slippage. For higher twists the strength decreases because of the torsional stress on the fibers. The filling

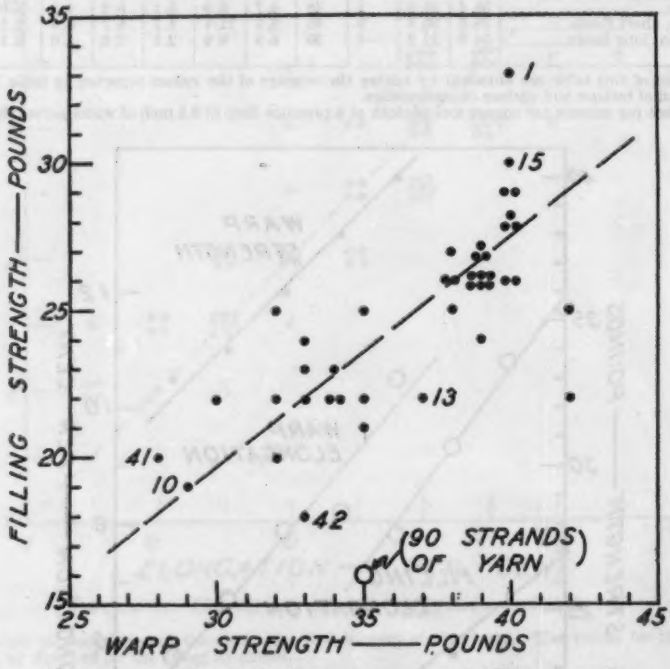

FIGURE 6.—*Correlation between warp and filling strength.*

The points plotted represent the warp and filling strength of the cloths. Weaves nos. 1 and 15 have a large number of thread interlacings compared to weaves nos. 10, 41, and 42.

yarns are weaker than the warp yarns because of the lower twist. This is reflected in the strength of the cloths. The filling strength is less than the warp strength for each weave.

The warp and filling strengths vary considerably with the weave. This is not due to variation in yarn strength, but to the manner of the interlacing of the two sets of yarn. If the manner of interlacing is regular then the strength increases with the number of interlacings per unit area because the stress is distributed more uniformly. This is shown qualitatively in figure 6 where the filling strength is plotted against the warp strength. Weaves nos. 1 and 15 represent those having a large number of interlacings while weaves nos. 10, 41, and

42 represent those having a small number of interlacings, see figures 1 and 3. Weave no. 13 has the longest floats and the cloth is very sleazy. This figure shows, as expected, that the warp and filling are affected similarly by the manner of interlacings.

The strengths of 90-warp and of 90-filling yarns by the multiple strand method are also plotted in figure 6. It is apparent that the filling strength of each cloth is much greater than the multiple strand strength of 90-filling yarns. This is not true for the warp. In fact for 13 cloths the warp strength is less than the multiple strand strength of 90-warp yarns. It is to be noted that these 13 cloths belong to the class having a small number of thread interlacings. The explanation of these observations is apparent when the twist of the yarn, the number of interlacings, and the chafing action of the reed and harnesses on the warp yarn are taken into consideration.

The filling yarns are weak because of the low twist. They are not weakened by chafing during weaving. Interlacing them with the warp yarns brings the cotton fibers of the yarn into closer contact. This effect increases with the number of interlacings. It is equivalent to increasing the twist in the yarn and produces an apparent increase in the strength of the filling yarn. This apparent increase in yarn strength is actually observed in the filling strength of the cloths and is commonly referred to as fabric assistance.

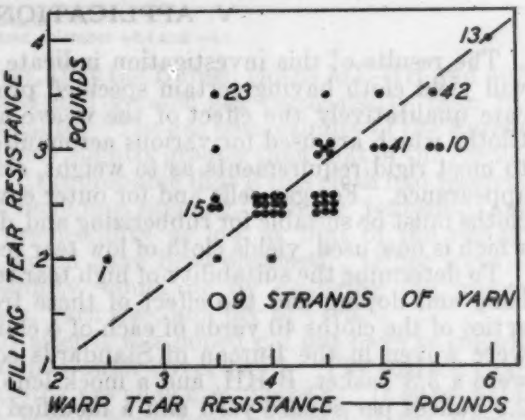

FIGURE 7.—*Correlation between warp and filling tear resistance.*

The points plotted represent the warp and filling tear resistance of the cloths. Weaves nos. 1 and 15 have a large number of thread interlacings and a firm texture. Weaves nos. 10, 13, 41, and 42 have a small number of thread interlacings and a sleazy texture.

The warp yarns are already relatively strong because of the high twist. They are weakened by chafing during weaving. Because of the high twist in the yarn the fibers are already in close contact with one another so that interlacing them with the filling yarns has relatively little or no effect. In fact, for those weaves having a small number of interlacings, the apparent increase in strength is not sufficient to balance the decrease due to chafing so that the fabric strength is actually less than the yarn strength. This may be referred to as negative fabric assistance. The fabric assistance observed for the weaves having a large number of interlacings is probably chiefly obtained as a result of greater uniformity of stress distribution.

The factors which contribute to the tear resistance of cloth are (1) the strength of the yarn perpendicular to the direction of the tear and (2) the freedom of movement of these yarns in the direction of the tear.

The importance of the first factor is obvious and is shown by the difference between the warp and filling tear (see table 1 and fig. 7).

The second factor contributes to high tear resistance because movement of the yarns, which are being torn, in the direction of the tear

distributes the tearing stress over several adjacent yarns. The greater the freedom of movement the greater will be the number of adjacent yarns which will carry the tearing stress and therefore the greater the tear resistance.

In figure 7 the warp tear is plotted against the filling tear. The warp tear is greater than the filling tear for every weave except no. 23. Weaves nos. 1 and 15 which have a large number of interlacings and a firm texture have the lowest tear resistance, while weaves nos. 10, 13, 41, and 42 which have a small number of thread interlacings, a sleazy texture, and long floats have the highest tear resistance. It is to be noted that the opposite was observed in the consideration of strength. As expected, the warp and filling are affected similiarly by the manner of thread interlacing. For comparison in figure 7 there are also plotted the equivalent strengths of 9 warp and of 9 filling yarns; that is, the tear resistance which would be obtained if the tearing stress were uniformly distributed over a cloth width equal to 0.1 inch.

V. APPLICATION

The results of this investigation indicate the type of weave which will yield cloth having certain specified properties. They also indicate qualitatively the effect of the weave upon the other properties. Cloths which are used for various aeronautical purposes usually have to meet rigid requirements as to weight, strength, permeability, and appearance. For gas cells and for outer cover cloth for dirigibles the cloths must be suitable for rubberizing and doping. The plain weave, which is now used, yields cloth of low tear resistance.

To determine the suitability of high tear resistance cloth for rubberizing and doping and the effect of these treatments upon the properties of the cloths 40 yards of each of 4 cloths of high tear resistance were woven in the Bureau of Standards cotton mill. These cloths were a 3/3 basket, B-HH, and a mock leno, ML-HH, each weighing 2.0 ounces per square yard and a modified mock leno, 40A, weighing 2.2 ounces per square yard to be rubberized similar to gas cell cloth and a 3/3 basket, B-BB, weighing 2.8 ounces per square yard to be doped similar to outer cover cloth for dirigibles.

The yarns used in these cloths were 160/2 spun from Sea Island cotton and were mercerized. They were the strongest yarns commercially obtainable of that size, strength being a factor which yields cloth of high tear resistance. The Bureau cloth 40A was given the regular balloon finish in a commercial finishing plant. Because of the small quantity of cloth and markedly different properties from that of the regular balloon cloth it was badly damaged in places, especially along the selvages, during the finishing process. The Bureau cloths B-HH, ML-HH, and B-BB were not finished but were rubberized and doped as they came from the loom.

These cloths were rubberized, doped, and tested by the Goodyear Zeppelin Corporation. The results are given in table 3. For comparison data are also given for the regular HH gas cell and regular BB outer cover cloths. The data show that the cloths woven at the Bureau of Standards are stronger than the cloths now used. The tear resistances of the Bureau cloths are 2 to 3 times those of the cloths now used. The Bureau cloths retain their superior strength and tear resistance after they are rubberized or doped.

TABLE 3.—*Effect of rubberizing and doping on the properties of cloth*

Designation	Description of cloth	Weight per sq. yd.	Breaking strength		Tear resistance		Gas permeability, liters per square meter in 24 hours
			Warp	Filling	Warp	Filling	
		Oz.	*Lb.*	*Lb.*	*Lb.*	*Lb.*	
HH	Regular gas cell cloth	2.0	[1] 38	[1] 38	3	2.7	
B-HH	Bureau of Standards cloth	2.0	47	43	8.8	12.6	
ML-HH	do	2.0	51	44	6.5	6.8	
40A	do	2.2	49	46	8.9	6.2	
HH	Regular rubberized gas cell cloth	5.8	[1] 40	[1] 40	1.8	1.8	Avg. 4.0. Max. 5.0.
B-HH	Bureau of Standards rubberized cloth.	5.7	57	50	7	4	Avg. 5.9. Max. 7.2. Min. 4.1.
ML-HH	do	5.9	55	47	3.5	3.5	Too high for test.
40A	do	5.8	56	46	11	7	First specimen 4.8. Second specimen 9.5. Third specimen too high for test.
BB	Regular outer cover cloth	2.9	[1] 60	[1] 60	4.8	3.2	
B-BB	Bureau of Standards cloth	2.8	66	67	9.6	9.4	
BB	Regular doped outer cover cloth	[2] 5.5	83	83	2.5	2.1	
B-BB	Bureau of Standards doped cloth	6.2	102	99	4.5	4.5	

[1] Minimum specified in existing standard.
[2] Nominal specified in existing standard, tolerance +0.4 and −0.2.

It is to be noted, however, that the Bureau cloths ML-HH and 40A after they were rubberized had greater gas permeability than the regular rubberized gas cell cloth. The gas permeability of the Bureau rubberized cloth B-HH is only slightly greater than that of the regular rubberized gas cell cloth. It is probable that if this cloth had received the regular balloon finish including singeing, desizing, tentering, and calendering, that a gas premeability as low as that of the regular rubberized gas cell cloth would have been obtained. Other ways of lowering the gas permeability would be by changing the weave, number of threads per inch, yarn size, and ply of yarn.

The Bureau cloth B-BB after it was doped did not have as smooth a surface as the regular doped outer cover cloth. The surface characteristics of the doped cloths are shown in figure 8. It is probable that a smoother surface would have been obtained if the Bureau cloth B-BB had received the regular balloon finish including singeing, desizing, tentering, and calendering. Other ways of obtaining a smoother surface would be by changing the weave, number of threads per inch, yarn size, and ply of yarn.

VI. SUMMARY

The effect of the weave on the properties of cloths of the same weight has been studied. In general a cloth which is closely woven, firm, and has a large number of threads interlacings per unit area and short floats has a greater strength, elongation, and take-up and has a lower tear resistance and air permeability than a cloth of the same weight which is loosely woven, sleazy, and has a small number of thread interlacings per unit area and long floats. The strength and elongation decrease as the tear resistance is increased. The factors which contribute to strength and tear resistance have been enumerated and discussed.

The effect of rubberizing and doping on the properties of cloth has been determined. Rubberizing and doping increased the strength and decreased the tear resistance and permeability. A cloth of initially high tear resistance retains its high tear resistance relative to a cloth of initially low tear resistance.

WASHINGTON, July 26, 1933.

U.S. Department of Commerce, Bureau of Standards

RESEARCH PAPER RP601

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

THE ISOTOPIC FRACTIONATION OF WATER

By Edward W. Washburn, Edgar R. Smith, and Mikkel Frandsen

ABSTRACT

When water is subjected to electrolysis an isotopic fractionation takes place, the evolved hydrogen being richer in the H^1 isotope and the evolved oxygen richer in the O^{16} isotope than is the water from which they are evolved. If the gases resulting from the electrolysis of water are recombined, the water produced has a lower density than the water from which they were evolved. The residual water grows progressively more dense as the elctrolysis proceeds, owing to the increasing concentration of the H^2 isotope of hydrogen and, to a less extent, also of the O^{18} isotope of oxygen. Curves illustrating the efficiency of the fractionation process are given. Substantially pure H^2, pure H^1, and pure O^{16} can be obtained by this fractionation process. The heavy water obtained by the process has a higher freezing point, a higher boiling point, and a lower refractive index than normal water. Isotopic fractionation of water by distillation and by adsorption has also been accomplished. A differential pycnometer method for measuring specific gravity, accurate to 1 part in a million, is described.

CONTENTS

		Page
I.	Introduction	453
II.	The preparation of water of low density	454
III.	The preparation of water of high density and the variation of density with continued electrolysis	454
IV.	The density measurements	457
V.	Purification of the water for density measurements	458
VI.	The relation between density and isotopic composition	458
VII.	Some physical properties of the heavy water	458
VIII.	Isotopic fractionation of water by distillation and by adsorption	461

I. INTRODUCTION

On December 9, 1931, an experiment was started at the Bureau of Standards for the purpose of attempting to produce an isotopic fractionation of water by electrolysis. In this experiment, normal water was acidified with sulphuric acid and electrolyzed between platinum electrodes.

The evolved gases were passed through a condenser for the purpose of condensing out the water vapor and collecting the spray and returning them to the system. The experiment was continued until 98 percent of the original water had been decomposed. The density of the residual water was then determined and found to be higher than that of normal water by 164 parts in a million. Before the completion of this experiment, samples of water were obtained from commercial electrolytic cells and examined spectroscopically and found to have a higher concentration of the H^2 isotope than normal water, thus proving that an isotopic fractionation of hydrogen takes place during electrolysis. This discovery was published [1] and it was stated

[1] Washburn, E. W., and Urey, Harold C., Proc. Nat. Acad. Sci., vol. 18, p. 496, July 1932. The density of one sample of the water which was examined spectroscopically was greater than that of normal water by 52 parts per million.

that the electrolysis was being continued for the purpose of determining the physical properties of the new kind of water and also of ascertaining whether an isotopic fractionation of oxygen was also taking place. The purpose of the present paper is to present the results thus far obtained. The experimental work here recorded is to be regarded as exploratory in nature and should be repeated at some future time with greater accuracy and with more attention to detail and to the effects of various factors which could not be studied in the initial experiments.

II. THE PREPARATION OF WATER OF LOW DENSITY

A solution of sodium sulphate in normal water was caused to flow continuously through an electrolytic cell at such a rate that 10 percent of it was decomposed by the electrolysis. The oxygen from this electrolysis was combined with commercial hydrogen, and the density of the resulting water measured and found to be less by 11 parts per million (two determinations, -10.6 and -11.0) than that of the water produced by burning in air hydrogen from the same tank, this latter water having the same density as normal water (one determination, -1 part per million). The hydrogen from the above electrolysis was also burned to water, the density of which was only slightly ($\gtrsim$ 5 parts per million) less than that of normal water.

The above experiment was then repeated with a larger amount of normal water which was decomposed only to the extent of 4 percent. The gases from this electrolysis were recombined and produced water having a density lower than that of normal water by 12 parts per million (two determinations, -12 and -13). This water was then re-electrolyzed in the same manner with 15 percent decomposition and the gases recombined. They produced water with a density lower than that of normal water by 19 parts per million (two determinations, -19.7 and, after redistillation, -18.8). This sample was tested for ammonia with Nessler's reagent with a negative result. A parallel experiment with water containing 0.2 part NH_3 per million gave a strong positive test.

The only plausible explanation of the above results appears to be the occurrence of an isotopic fractionation of oxygen, electrolytic oxygen being richer in the O^{16} isotope than the water from which it is generated. By a systematic continuation of experiments of the above character, it should be possible to prepare water of the formula $H_2^1O^{16}$ and consequently H^1 hydrogen and O^{16} oxygen. With these pure gases available in quantity, it will be possible to prepare water composed of H^1 hydrogen combined with normal oxygen and water composed of O^{16} oxygen combined with normal hydrogen. On electrolysis, the first of these will fractionate only with respect to oxygen and the second only with respect to hydrogen.

III. THE PREPARATION OF WATER OF HIGH DENSITY AND THE VARIATION OF DENSITY WITH CONTINUED ELECTROLYSIS

Fifteen liters of water having an initial specific gravity of 1.000034 was obtained from a commercial electrolytic cell.[2] This water was made 0.01 *N* with sulphuric acid and was then subjected to electrolysis

[2] We are indebted to the Southern Oxygen Company of South Washington, Va., for this water.

with a current of 21 amperes between an anode of bright platinum and a gold-plated copper cathode covered with platinum black.

From time to time the electrolysis was interrupted and the specific gravity of the residual water determined. During the first part of the electrolysis, the excess of specific gravity (that is, the excess over that of normal water taken as unity) was doubled each time the volume of the water was reduced one half. The complete specific gravity curve is shown in figures 1 and 2.

When the volume of the residual water had been reduced to 4 liters, it was temporarily transferred to an electrolytic apparatus so

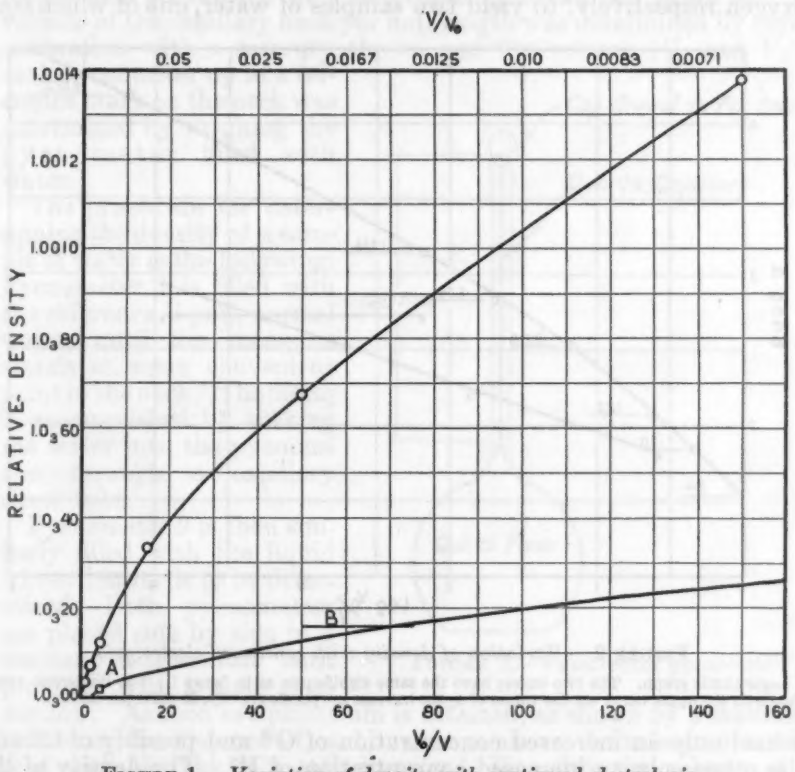

FIGURE 1.—*Variation of density with continued electrolysis.*

The ordinates are values of the specific gravity, the abscissae values of the ratio of the initial volume of the water to its volume at the time the current was interrupted in order to determine the density. The upper curve represents the density of the residual water, the lower curve that of the water formed by recombining the evolved gases.

designed as to permit the collection of the two gases separately. The oxygen evolved from this water was combined with normal hydrogen to produce 300 ml of water which was found to have normal density. The hydrogen was combined with normal oxygen and the specific gravity of the resulting water was measured and is represented on figure 1 by point *A*. Again, when the volume of the residual water had reached 300 ml ($D=1.000674$), the evolved gases were recombined to produce 100 ml of water whose specific gravity (1.000161) is represented on figure 1 by point *B*. The locus of the curve through points A and B when compared with the density curve of the residual

water shows that a high efficiency of fractionation was being maintained as the electrolysis proceeded.

When the specific gravity of the residual water had reached 1.001376, the electrolysis was interrupted in order to measure some of the physical properties of this heavy water. These are reported below.

When electrolysis was resumed the water was made conducting with dehydrated barium hydroxide [3] and electrolyzed between platinum electrodes until its volume had been reduced to 18 ml. During this stage of the electrolysis, the oxygen and hydrogen evolved were dried and separately combined with normal hydrogen and normal oxygen respectively, to yield two samples of water, one of which con-

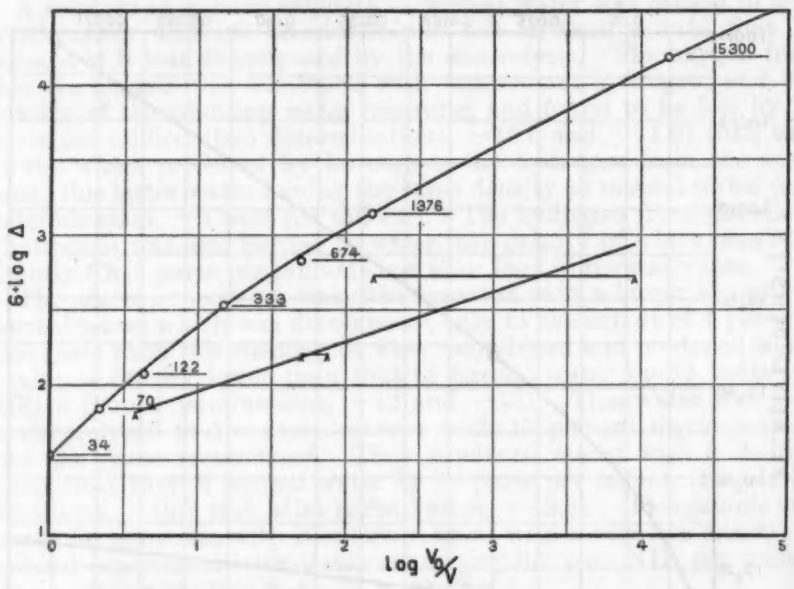

FIGURE 2.—*Variation of density with continued electrolysis.*

Logarithmic graph. The two curves have the same significance as in figure 1. The numerical values shown on the upper curve are the values of Δ, the increase in specific gravity, in parts per million.

tained only an increased concentration of O^{18} and possibly of O^{17} and the other only an increased concentration of H^2. The density of the first of these lots of water was measured and found to be about 18 parts per million higher than that of normal water. This increased density is further evidence of an isotopic fractionation of oxygen[4] but, as would be expected, the efficiency of the oxygen fractionation is much less than that of the hydrogen.

The specific gravity of the water produced by burning the hydrogen was also measured and found to be 1.000539. The locus of this value is shown on figure 2.

The electrolysis was continued until a final sample of water 2.1 ml in volume was obtained. This water had a specific gravity of 1.0153. During this part of the electrolysis, the hydrogen evolved was col-

[3] The addition of barium oxide for the purpose of giving a conducting solution has the advantage that a constant concentration of electrolyte can be maintained during the electrolysis owing to the limited solubility of $Ba(OH)_2 \cdot 8H_2O$, and the water held by this compound is readily recovered by ignition or by treating the solution with carbon dioxide and filtering off the barium carbonate formed. By this procedure none of the hydrogen is retained by the electrolyte.

[4] See p. 454.

lected and, after purification, its atomic weight was determined by reduction of CuO and found to be 1.012.

IV. THE DENSITY MEASUREMENTS

Since the course and extent of the isotopic fractionation are most readily measured by the change in the density of the water, it was necessary to have available an accurate method for measuring small density differences. The following method was devised for this purpose.

The density measurements were made with twin pycnometers of fused silica provided with 1 mm capillary necks (see figure 3). The volume of the capillary neck per unit length was determined by careful calibration with a mercury thread and the volume (V_1 and V_2) of each pycnometer up to a reference mark on the neck was determined by weighing the pycnometer filled with water.

The procedure for determining the density of a sample of water is the following: Pycnometer 1 is filled with the reference liquid, normal water, until the meniscus stands at some convenient point in the neck. The filling is accomplished by sucking the water into the pycnometer through a capillary silver tube.

Pycnometer 2 is then similarly filled with the liquid whose density is to be determined. Both pycnometers are placed side by side in a constant temperature bath provided with a plate glass window. As soon as equilibrium is attained, as shown by a stationary meniscus, the distance of each meniscus above the reference mark is read with the aid of a cathetometer.[5] Knowing the volume of the capillary per unit length, the difference $\Delta C = \mu_1 - \mu_2$, in the masses of the water above the reference marks in the two pycnometers, 1 and 2, can be computed.

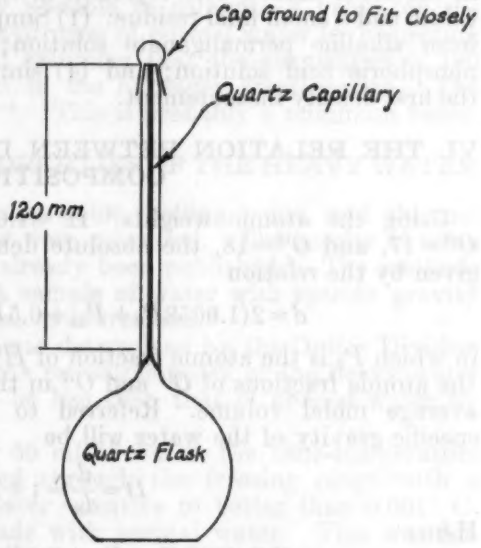

FIGURE 3.—*Fused-silica pycnometer*

The caps are now placed on the pycnometers and these are dried, placed on opposite pans of a balance and the difference, $\Delta m = m_1 - m_2$, in their masses determined to the nearest 0.03 mg. The above series of operations is now repeated with the reference liquid in pycnometer 2 and the other liquid in pycnometer 1, giving respectively $\Delta C'$ and $\Delta m'$.

The relative densities of the two liquids are then calculated from the relation

$$D = \frac{d}{d_N} = 1 + \frac{\Delta m - \Delta m' - \Delta C + \Delta C'}{d_N(V_1 + V_2)} = 1 + \Delta \qquad (1)$$

where d_N is the density of the reference liquid, in this instance water.

[5] Or, with some sacrifice of accuracy, by means of graduations on the neck of the pycnometer.

Obviously, it is not necessary to make any corrections for reduction to vacuo in any case where this method would be used, that is, where d is approximately equal to d_N. The method is essentially a differential one and with 60 ml pycnometers the results are reproducible to about 1 part in a million.

V. PURIFICATION OF THE WATER FOR DENSITY MEASUREMENTS

All of the density measurements reported in this paper were made on samples of carefully purified water. The method of purification followed was essentially that employed in preparing high-grade conductivity water. It consisted in the following series of distillations in the order named with rejection in each case of a small initial distillate and a small final residue: (1) Simple distillation; (2) distillation from alkaline permanganate solution; (3) distillation from dilute phosphoric acid solution; and (4) simple distillation, usually after the first density measurement.

VI. THE RELATION BETWEEN DENSITY AND ISOTOPIC COMPOSITION

Using the atomic weights,[6] $H^1=1.00778$, $H^2=2.01363$, $O^{16}=16$, $O^{17}=17$, and $O^{18}=18$, the absolute density of a sample of water is given by the relation

$$d=2(1.0058P_2+P_{18}+0.5P_{17}+9.00778)/V \tag{2}$$

in which P_2 is the atomic fraction of H^2 in the hydrogen, P_{18} and P_{17} the atomic fractions of O^{18} and O^{17} in the oxygen and V the effective average molal volume. Referred to normal water as unity the specific gravity of the water will be

$$D=\frac{d}{d_N}=1+\Delta. \tag{3}$$

Hence

$$\Delta=\frac{2(1.0058P_2+P_{18}+0.5P_{17}+9.00778)-Vd_N.}{Vd_N} \tag{4}$$

For normal water, if we put

$$P_2=(3\pm 2)\times 10^{-5} \tag{5}$$

$$P_{18}=(159\pm 3)\times 10^{-5} \tag{6}$$

$$P_{17}=\text{negligible} \tag{7}$$

we have

$$Vd_N=2(1.0058P_2+P_{18}+9.00778) \tag{8}$$

$$=18.0188\pm 0.0001. \tag{9}$$

Hence, if V is independent of the isotopic composition,

$$\Delta=\frac{2.0116P_2+2P_{18}+P_{17}-(0.0032\pm 0.0001)}{18.0188\pm 0.0001}. \tag{10}$$

If in the given sample of water, the oxygen is normal oxygen, equation (10) gives

$$P_2=8.957\Delta+0.00003\pm 0.00008. \tag{11}$$

[6] Bainbridge, Kenneth T., Phys. Rev., vol. 44, p. 57; 1933.

If the hydrogen is normal hydrogen, equation (10) gives

$$P_{18}+0.5P_{17}=9.0094\Delta+0.0016\pm0.00007. \quad (12)$$

The calculation of P_2 or of $P_{18}+0.5P_{17}$ by the above equations is rendered uncertain to the extent indicated by the ± signs, owing to our present lack of exact knowledge of the isotopic compositions of normal hydrogen and normal oxygen. When pure H^1 and pure O^{16} become available (and their preparation appears to offer no particular difficulty) it will be possible to obtain exact equations for P_2 and for $P_{18}+0.5P_{17}$ in terms of Δ, provided the molal volume V can be shown to be independent of the isotopic composition.

From the data obtained in our first experiment, described in section 1 above, it is obviously possible to compute a value for the atomic fraction of H^2 in the hydrogen of normal water by neglecting the oxygen fractionation and assuming that the electrolysis is 100 percent efficient with respect to the hydrogen fractionation. The value thus obtained is 3×10^{-5}. This is probably a minimum value.

VII. SOME PHYSICAL PROPERTIES OF THE HEAVY WATER

The refractive index, freezing point, boiling point, and thermal expansion of the heavy water were measured. The results of some of these measurements have already been published.[7] The methods used are described below. A sample of water with specific gravity 1.001376 was used in all of the measurements.[8]

The refractive index.—This was determined by the Optics Division of this Bureau. At 25° C, the refractive index of the heavy water for $\lambda=5876$ A was found to be less than that of normal water by $(59\pm1)\times10^{-6}$.

The freezing point.—Using 60 ml of water, the time-temperature cooling curve was determined through the freezing range with a platinum resistance thermometer sensitive to better than 0.001° C. The first experiment was made with normal water. This was followed by two runs with the heavy water and these by a second run with normal water. The results are shown in figure 4. The flatness of the curves in the freezing range indicates that during crystallization isotopic fractionation does not occur to an appreciable extent for the volume of water used in these experiments.

The boiling point.—Twin vacuum-jacketed boilers of the Cottrell[9] type provided with internal coil heaters of platinum were connected by a 10-junction thermel of copper-constantan. The difference in temperature of the two junctions was measured with a Brooks-Spinks[10] potentiometer and a sensitivity of better than 0.001° was attained. In the first experiment, normal water was placed in both boilers and the zero of the apparatus determined. The normal water in one boiler was then replaced by the heavy water and the difference in boiling temperatures directly measured. The two kinds of water were then interchanged in the two boilers and the experiment repeated. The above set of experiments was then repeated with a White poten-

[7] Washburn, Edward W., Smith, Edgar R., and Frandsen, Mikkel, J. Chem. Physics, vol. 1, p. 288, 1933.

[8] Later measurements with a sample of water of specific gravity 1.1 have recently been reported by G. N. Lewis, J. Am. Chem. Soc., vol. 55, p. 3057, 1933.

[9] Cottrell, F. G., J. Am. Chem. Soc., vol. 41, p. 721, and figure 1, p. 730, 1919.

[10] Brooks, H. B., and Spinks, A. W., Bur. Standards Jour. Research, vol. 9, p. 781, 1932.

tiometer. All four results agreed and showed that the boiling point of the heavy water was higher than that of normal water by $0.02 \pm 0.002°$ C.

The temperature of maximum density.—The twin silica-glass pycnometers were filled, one with normal water and the other with the heavy water, and placed side by side in the regulated bath. The bath was held at a series of constant temperatures between 2.5 and

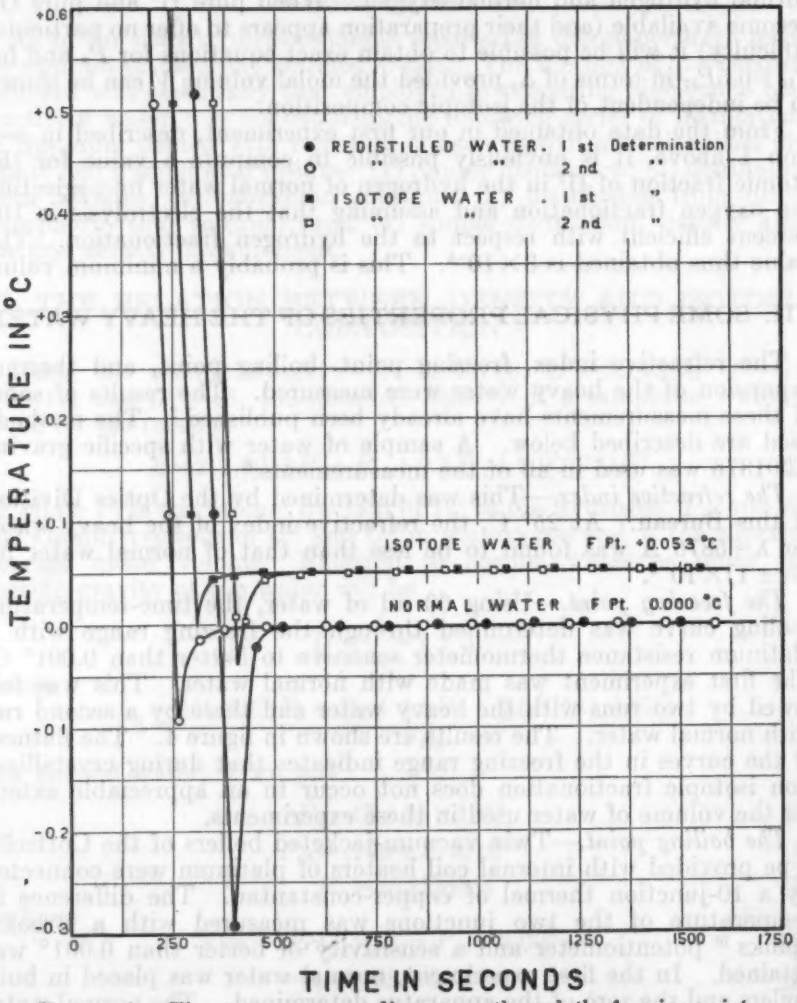

FIGURE 4.—*Time-temperature cooling curves through the freezing point.*
The "isotope water" employed had a specific gravity of 1.001376.

4.9° C. and when equilibrium was attained the volume of each sample of water was measured by determining with a cathetometer the position of the meniscus in the capillary. The relation between temperature and volume is displayed graphically in figure 5. The peculiar effect observed in the neighborhood of 3.75° is apparently real but discussion of this phenomenon will be postponed until further measurements can be made.

VIII. ISOTOPIC FRACTIONATION OF WATER BY DISTILLATION AND BY ADSORPTION [11]

By distilling with the 35-foot packed-column still described in a previous publication,[12] two fractionations were made with the results shown in table 1.

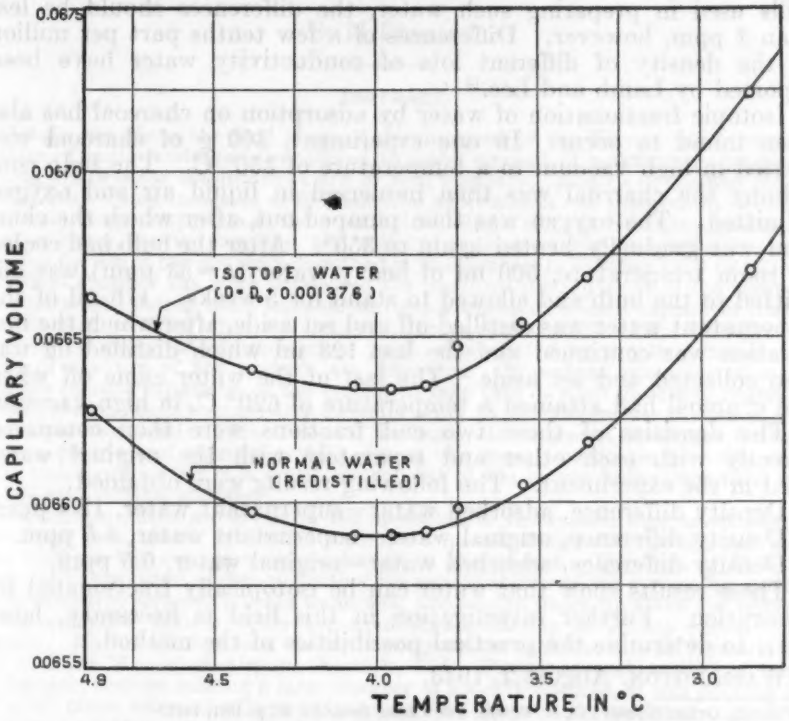

FIGURE 5.—*Thermal expansion of water in the neighborhood of 4° C.*

TABLE 1

[ppm=parts per million]

Water used	Volume distilled	Date of distillation	Density increase		Density difference directly determined. Residue-distillate
			Still residue	Initial distillate	
	l		*ppm*	*ppm*	*ppm*
Normal Δ=0	1.5	December 1931	32	−14	41
Heavy Δ=53.3 ppm	10	April 1933	53.3	−13.2	64.9

These results demonstrate that isotopic fractionation by distillation is entirely feasible and this process should find application as a supplement to fractionation by electrolysis. Further investigation is needed in order to determine how distillation operates with respect to the fractionation of oxygen and of hydrogen, since it depends

[11] The results of these experiments were briefly reported in a previous publication. Washburn, Edward W., and Smith, Edgar R., J. Chem. Phys., vol. 1, p. 426, 1933.
[12] Bruun, Johannes H., and Schicktanz, Sylvester T., Bur. Stands. J. Res., vol. 7, fig. 17, p. 872, 1931.

upon a different principle and may be more efficient, for example in the fractionation of the oxygen isotopes, than is the process of electrolysis.

Ordinary conductivity water as prepared in the laboratory is the result of several distillations and different lots of such water may be expected to show small differences in density. Owing to the inefficient stills used in preparing such water, the differences should be less than 1 ppm, however. Differences of a few tenths part per million in the density of different lots of conductivity water have been reported by Lamb and Lee.[13]

Isotopic fractionation of water by adsorption on charcoal has also been found to occur. In one experiment, 300 g of charcoal was heated in high vacuum to a temperature of 350° C. The bulb containing the charcoal was then immersed in liquid air and oxygen admitted. The oxygen was then pumped out, after which the charcoal was gradually heated again to 350°. After the bulb had cooled to room temperature, 500 ml of heavy water ($\Delta = 53$ ppm) was admitted to the bulb and allowed to stand for 5 weeks. 176 ml of the supernatant water was distilled off and set aside, after which the distillation was continued and the last 123 ml which distilled off was also collected and set aside. The last of the water came off when the charcoal had attained a temperature of 620° C. in high vacuum.

The densities of these two end fractions were then compared directly with each other and separately with the original water used in the experiment. The following results were obtained:

Density difference, adsorbed water − supernatant water, 12.4 ppm.
Density difference, original water − supernatant water, 6.5 ppm.
Density difference, adsorbed water − original water, 6.5 ppm.

These results show that water can be isotopically fractionated by adsorption. Further investigation in this field is necessary, however, to determine the practical possibilities of the method.

WASHINGTON, August 2, 1933.

[13] Lamb, Arthur B., and Lee, R. Edwin, Am. Chem. Soc., vol. 35, p. 1681, 1913.

U.S. Department of Commerce, Bureau of Standards

RESEARCH PAPER RP602

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

PERFORMANCE TESTS OF RADIO SYSTEM OF LANDING AIDS

By H. Diamond

ABSTRACT

Details are given the final stage of development of the Bureau of Standards system of radio landing aids designed to assist aircraft in making safe landings during fog or other conditions of low or zero visibility. Results of the first stage of the work which consisted of fundamental experiments and research to develop the basic component parts of the system, including a runway localizing beacon, a landing beam, marker beacons, and suitable radio receiving and indicating apparatus for use in the air, were described in this Journal in October 1930 (RP238). The final stage of the work comprised the practical development of the component parts and fitting them together to form a workable system capable of ready coordination with existing and projected aeronautical radio aids. Changes in engineering design of the component parts, necessitated by practical considerations of the use of the system, are described. Important examples of such changes include expansion of the system to take care of wind direction, redesign of the landing beam transmitting antenna array to provide greater control of the horizontal and vertical directivity of the beam, development of a more flexible marker beacon and antenna system, design of a more practicable receiving system, and coordination in a single instrument of the course indications used for lateral and vertical guidance.

Performance data are given on the operation of the system over an extended period of time, which indicate its complete practicability for commercial use. The tests on the system were conducted in two phases. The first, at College Park, Md., consisted of demonstrating the practicability of the system by means of an extensive series of hooded landings. The second phase involved the testing of the complete system under the conditions obtaining at a busy commercial airport. An installation was made, in cooperation with the city of Newark, at the Newark Municipal Airport, Newark, N.J. During several months of tests at Newark, besides making a large number of hooded landings, it was possible to fly at all times when all other airplanes were on the ground because of fog. The operation of the system was demonstrated in the air to many engineers and officials as well as to nearly one hundred air transport pilots. Perhaps the most striking demonstration consisted of a completely blind flight from College Park, Md., to the Newark airport, during which radio was the sole means used for directional guidance and for landing.

CONTENTS

	Page
I. Introduction	464
II. General description of the system	464
III. Practical application of the system	466
1. Runway localizing beacon	466
2. Landing beam	470
3. Marker beacons	479
4. Monitoring and communication system	481
5. Coordination of receiving and indicating equipment	482
IV. Operation of the system	483
1. Method of making a landing during fog	483
2. Use of system in event of failure of one of its elements	486
3. Simplification of system for economy purposes	487
V. Performance data and tests	487
1. Tests at College Park, Md	487
2. Demonstrations and tests at Newark, N.J	488
VI. Acknowledgements	490

I. INTRODUCTION

Research work on a radio system for landing aircraft during zero visibility, carried on in the Research Division of the Aeronautics Branch of the Department of Commerce, at the Bureau of Standards, during 1928 to 1930, was described in the October 1930 issue of this Journal.[1] The work covered the first stage of development of the system, comprising fundamental experiments and research to develop tools for attacking the problem. The runway localizing beacon, the landing beam, and the marker beacon, constitute these tools. The present paper describes the final stage of the development, consisting of improvement of these tools; fitting them together to form a complete and workable system at both the transmitting and receiving ends; and, finally, demonstrating the practicability of the system by means of hooded landings and by flights and landings at a commercial airport under conditions of visibility causing complete interruption of all other flying.

II. GENERAL DESCRIPTION OF THE SYSTEM

Prior to analyzing the results achieved during the final stage of this project, it is desirable to review briefly the problems incidental to the "instrument" landing of aircraft and how the radio system of landing aids provides a solution to these problems. To land an aircraft safely at an airport completely blanketed by fog, it is necessary for the pilot, (*a*) to find the vicinity of the airport, and (*b*) to find and reach a suitable point of landing. The radio range-beacon system now in use on the civil airways of the United States renders the solution of the first phase of the problem an accomplished fact. The pilot following the beacon signals is guided directly over the beacon station (which is located within a few miles of the airport), and thereby learns his exact position with respect to the airport. The second phase of the problem requires information on the position of the landing aircraft in three dimensions. Lateral and longitudinal guidance are required to determine the direction of landing and the boundaries of the landing field, while vertical guidance is necessary for the determination of altitude and of the freedom of the landing path from obstacles. In the system of radio landing aids three elements are utilized to give the necessary guidance in three dimensions.

The runway localizing beacon gives indications of the lateral position of the aircraft with respect to the airport and permits keeping the aircraft directed to and over the desired landing runway. A 200-watt transmitting set of the visual beacon type, operating in the beacon range of frequencies (200 to 400 kc) and feeding two small, multi-turn loop transmitting antennas, is employed. One of the beacon courses produced is oriented to coincide with the desired landing direction, depending on the wind conditions. On the aircraft the receiving set normally used by air transport operators for the reception of radio range-beacon signals and airways weather broadcasts is employed for receiving the runway-beacon signals. This set is supplemented by a reed converter [2] to convert the beacon signals to

[1] H. Diamond and F. W. Dunmore, A Radiobeacon and Receiving System for the Blind Landing of Aircraft. B.S. Jour. Research, vol. 5 (RP238), page 897, October 1930.

[2] F. W. Dunmore, A Course Indicator of Pointer Type for the Visual Radio Range-Beacon System. B.S. Jour. Research, vol. 7 (RP 336), p. 147, July 1931; Proc. I.R.E., vol. 19, p. 1579, 1931.

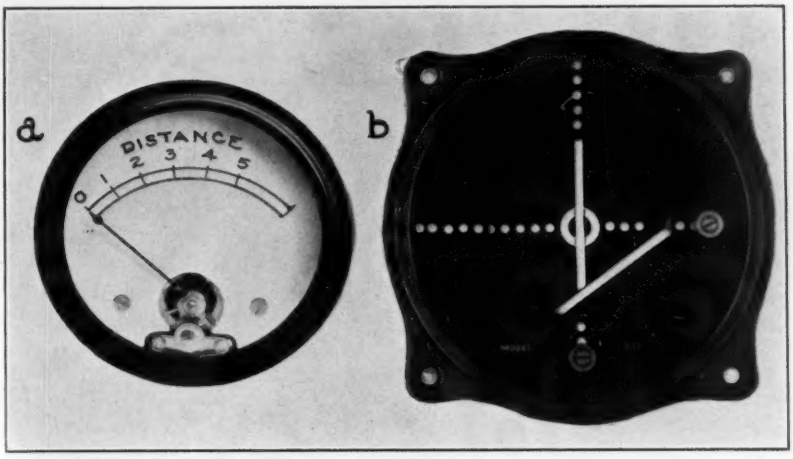

FIGURE 1.—*Indicating instruments required on the aircraft for utilizing the radio system of landing aids.*

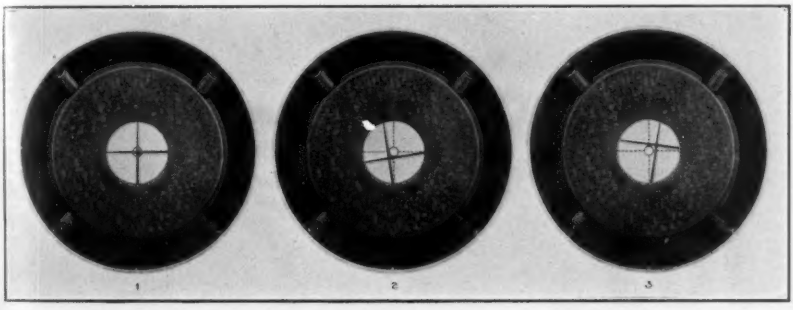

FIGURE 2.—*Typical course indications with combined instrument.*

1, Aircraft on proper spatial landing path; 2, aircraft to the left of desired landing direction and too low; 3, aircraft to the right of desired landing direction and too high.

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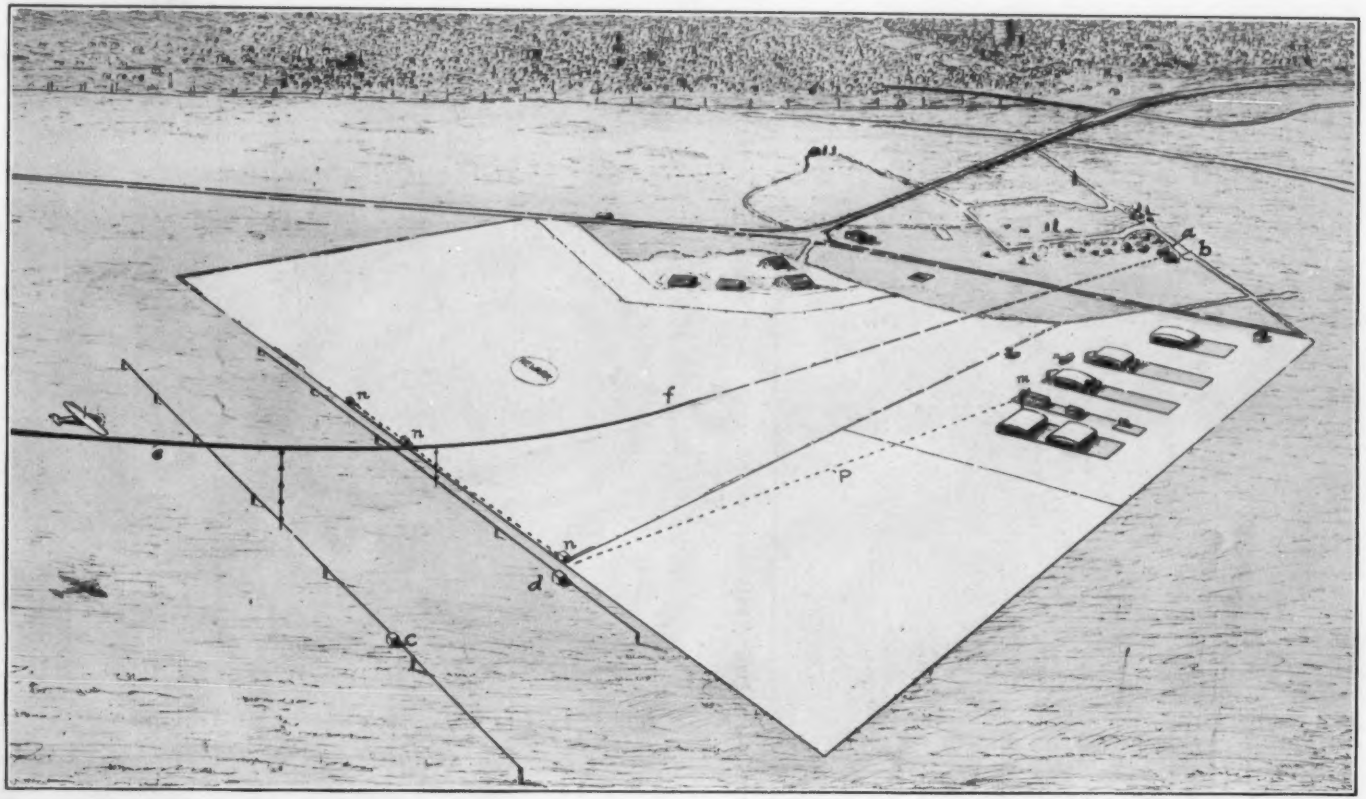

FIGURE 3.—*Three-dimensional view showing layout of ground transmitting equipment in the radio landing installation at the Newark Municipal Airport.*

a, Landing beam station; *b*, runway localizing beacon; *c*, approach marker beacon; *d*, boundary marker beacon; *e*, spatial landing path; *f*, point of contact with ground; *m*, location of monitoring and 2-way communication equipment; *n*, remote control stations for 2-way communication; *p*, control cable.

pointer type course indications, and also by an automatic volume control unit [3] whose function is to relieve the pilot of the burden of continuously adjusting the sensitivity of the receiving set as the distance between the aircraft and the ground station changes. The course indicator consists of the vertical pointer of a combined instrument shown in figure 1 (*a*). This pointer is pivoted about its lower end and swings left or right of a vertical index line depending upon whether the aircraft is on one side or the other of the runway course. A reversing switch is provided so that the deflection of the pointer and the direction of deviation of the aircraft coincide whether the aircraft is flying *away from* or *toward* the beacon.

Vertical guidance of the aircraft is given by a horizontally polarized ultra-high-frequency landing beam directed at a small angle above the horizontal and used in such a way as to provide a very convenient gliding path for the landing aircraft. The frequency of operation is 90,800 kilocycles (3.3 meters).

On the aircraft a simple ultra-high-frequency receiver is used, fed by a transmission line from a horizontal half-wave receiving antenna which is located in the wing slightly ahead of the leading edge. The rectified output from this receiving set operates the horizontal pointer of the combined instrument shown in figure 1 (*a*). The receiver sensitivity is so adjusted that the line of constant received signal below the inclined axis of the beam, corresponding to half-scale deflection of the horizontal pointer, marks out a landing path which is suitable for the aircraft and airport considered. The horizontal index line across the face of the combined instrument represents the point of half-scale deflection and corresponds to the proper landing path. The horizontal pointer represents the position of the aircraft relative to this path. A rise of this pointer above the horizontal index line indicates that the aircraft is above the proper landing path, while the reverse is true if the pointer falls below the index line.

Consideration of the operation of the combined instrument will show that the point of intersection of the two pointers represents the position of the aircraft relative to the desired landing runway and the proper landing path. Figure 2 shows three typical readings on the combined instrument. The coordination of the two sets of course indications into a single reading is of utmost importance to the pilot, relieving him of the need for considerable mental effort. Deviations from both courses may be corrected simultaneously. By keeping the pointers crossed over the small central circle on the instrument face, a suitable spatial landing path is followed down to the point of landing.

Longitudinal position of the aircraft as it approaches the airport is given by the combination of a distance indicator on the aircraft with the aural signals received from two marker beacons. The distance indicator, see figure 1 (*b*), consists simply of a direct-current milliammeter connected in the plate supply to the radio-frequency amplifying tubes of the beacon receiving set. Since the automatic volume control operates to increase the negative biasing voltage on the grids of these tubes with increasing input voltages to the receiving set, the plate current is approximately inversely proportional to the field intensity of the runway beacon. The instrument may therefore be calibrated in miles from the beacon (say, 0 to 5 miles). The

[3] W. S. Hinman, Jr., Automatic Volume Control for Aircraft Radio Receivers. B.S. Jour. Research, vol. 7 (RP 330), p. 37, July 1931.

distance indication secured is approximate only, but is sufficiently accurate for all necessary maneuvers of the landing aircraft at distances from the airport of the order of 1 to 5 miles.

Absolute indication of the longitudinal position of the aircraft when near the airport is given by aural signals from two low-power marker-beacon transmitters. One marker beacon is located about 2,000 feet from the approach end of the airport while the other marker defines the boundary or edge of the landing field. Different modulation frequencies are employed for the two marker beacons to facilitate ready identification of the one being passed over; the marker beacon at the field boundary having a modulation of about 250 cycles and the approach marker beacon a modulation of 1,250 cycles. The marker beacon transmitting antennas provide for great flexibility of operation; any portion of the landing field boundaries and approaches may be defined, and any radio frequency may be used in the range of from 200 to 20,000 kilocycles. These features will be discussed in more detail in section III.

III. PRACTICAL APPLICATION OF THE SYSTEM

The foregoing description outlines the general principles underlying the operation of the system. A number of practical problems arise in the application of these principles to provide a workable transmitting and receiving set-up. These problems have influenced to considerable extent the engineering design of the component elements of the system. To facilitate discussion of the engineering factors involved, repeated reference will be made to the experimental installation of the landing system at the Newark Municipal Airport, Newark, N.J. A three-dimensional view showing the layout of the ground transmitting equipment at Newark is given in figure 3.

1. RUNWAY LOCALIZING BEACON

The question of location at a given airport of the runway localizing beacon transmitter and antennas is related to the number of landing directions it is desired to serve, which, in turn, depends upon the prevailing wind directions during conditions of low visibility. If it is desired to provide for all wind directions so that an aircraft may land into the wind under all conditions, several expedients are available.

One arrangement is to mount the transmitter, antennas, and power supply, on a truck and to furnish service into the wind by moving the truck to the proper position just off the field. This method is not economical since special design features are required in the equipment. Moreover, a major objection is that the runway beacon service is interrupted between changes of truck location. Such factors as snow, ice, tire trouble, engine trouble, traffic, and inaccessibility of the truck position corresponding to certain wind directions, may make this interruption somewhat lengthy.

A second arrangement is to locate the loop transmitting antennas in the center of the airport in a pit provided with sturdy roof construction level with the surface of the airport so that an airplane may roll over it. The transmitter may also be located in the pit or may preferably be installed in a location more convenient for maintenance and operation. In the latter case, radio-frequency power is fed to the loop antennas by means of buried transmission-line cables. The

installation may be operated as a 4-course beacon with the four courses aligned to coincide with four major runway directions, or it may be used as a beacon giving two 180° courses, one of which may be oriented into the wind at all times, either by remote control from the operations office or automatically. The operation of the runway beacon underground has been tested and found satisfactory. An experimental pit installation was made at College Park for these tests. The distance range was reduced by about 50 percent from that obtained with the runway beacon above ground; the courses were found to be well defined and somewhat sharper than for above-ground operation.

A third arrangement is to place the runway beacon in any convenient location and to provide a buried transmission-line system whereby radio-frequency power may be fed to one or the other of two loop-antenna systems, each located just off the airport in line with a major runway direction. Transmission lines of the order of several thousand feet in length may be employed without undue loss of power. By strategically locating the two sets of loop antennas, practically all wind directions may be served. This method provides the most economical and efficient means for taking care of all wind directions.

At most airports, it is not expected that it will prove necessary to serve more than one landing direction. A study of meteorological data for a number of airports has shown that the wind during low visibility conditions generally comes from one major direction. The following data summarizes the conditions for the Newark airport during the period July 1, 1930, to June 30, 1931. Ceilings under 1,000 feet were observed 740 times; the wind was calm 25 times; north 128 times; northeast 291 times; east 107 times; southeast 67 times; south 34 times; southwest 57 times; west 16 times; northwest 15 times. Thus, for all ceilings below 1,000 feet, the prevailing wind direction was from the northeasterly quadrant. Accordingly, the runway beacon is located at the northeast end of the Newark airport and one of the courses is oriented along the northeast-southwest runway. In this installation, the loop antennas are housed in the same building as the transmitting set to provide protection from the elements. Each antenna consists of 7 turns of no. 10 wire wound on a wooden frame 12 feet long by 10 feet high, with a 2-inch spacing between turns. A goniometer is provided to swing the course anywhere between the two hangar lines, thereby giving service over a sector of approximately 40°.

To secure a true picture of the percentage of time, during low visibility, when the Newark runway beacon is useful, it is necessary to consider two other factors in addition to the data on wind directions given in the foregoing paragraph. The first is that for ceilings above 100 feet, the landing system may be used regardless of wind direction. A pilot breaking through this ceiling finds himself at the southwest approach to the field; from that point he may maneuver the aircraft (underneath the ceiling) to land in any desired direction. The second factor is that, as the ceiling becomes progressively lower than 1,000 feet, the wind velocity becomes correspondingly lower so that, finally, at ceilings of 100 feet or lower, the wind velocity rarely exceeds 6 to 8 miles per hour. Under these conditions, landings may be made downwind or crosswind if required. It is thus evident that a 1-direction runway beacon service, if properly oriented, is sufficient for all

practical purposes. As a matter of fact, during several months of tests at Newark, it was found unnecessary to use the goniometer for swinging the course in the 40° sector between the hangar lines. Fixed orientation along the northeast-southwest runway was found preferable, since the fixed courses and quadrants could then be employed for fixing definitely the position of the aircraft with respect to the airport during blind flights. In the case of airports located at a considerable distance from airway radio range beacons, this constitutes an important feature in finding the airport.

Other details to be considered in connection with the runway localizing beacon are: radio frequency; required sharpness of the course; and the effect of power lines, railroad tracks, and the like, upon the straightness of the course. The radio frequency on which the runway beacon operates should be at one end or the other of the beacon band, so that its signals may be received on the beacon receiving set carried aboard aircraft and at the same time a minimum of interference be had from nearby 2-kilowatt airway radio range beacons. The Federal Radio Commission has proposed setting aside the frequency, 224 kilocycles, for this service at all airports; this frequency fulfills the above requirements.

In the course of prolonged tests on the system, it was found desirable to provide a very sharp runway beacon course in order to keep the aircraft as accurately as possible along the desired landing direction and thereby to clear obstructions in the approach to the airport. This was particularly true in the experiments at College Park, Md., where the approach is quite restricted. The width of a radiobeacon course becomes progressively less as the beacon is approached. When the course was adjusted to the desired sharpness at a few miles from the airport, it became quite difficult for the pilot to follow the course at close range to the beacon. Once off the course, an attempt to get back on would result in overcompensation or "hunting" about the course, which would become progressively worse until finally the pilot could no longer follow the course. Two means were adopted to overcome this difficulty. The first consisted of utilizing a directional receiving antenna on the aircraft, the directivity being secured through the addition to the usual vertical pole antenna of a flat-top element running back to the vertical rudder fin. See figure 4. The antenna is then equivalent to an inclined receiving antenna with backward inclination. The directional effect is observed when flying at an angle to the course. Upon turning into the course there is an apparent moving of the course toward the airplane. The reverse is true when turning away from the course. The net result is to provide automatic compensation for the pilot's tendency to "hunt" or "weave" about the course. The second means adopted was to use the vertical pointer of the combined instrument simply as a check instrument, the primary instrument depended upon to determine the directional heading of the aircraft being the compass, preferably of the gyroscopic type. When the vertical pointer shows that the aircraft has deviated from the runway course, the compass bearing is altered to provide gradual return to the course. After several corrections, the proper compass bearing is determined which will provide just the proper angle of "crab" into the wind to maintain the aircraft along the desired course. It is important to note that the angle of correction for wind drift is thereby also determined, this angle being equal to the difference

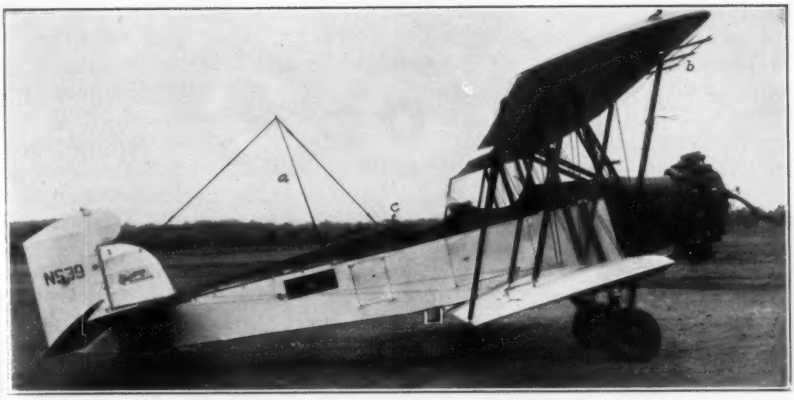

FIGURE 4.—*Airplane used in landing tests.*

a, Beacon receiving antenna; *b*, landing beam receiving antenna; *c*, hood for blind landings.

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between the compass bearing required to keep the aircraft on the runway course minus the true compass bearing of the runway. The use made of this correction angle will be described in section IV.

A direction finder on the aircraft, with bilateral pointer-type course indication, has been suggested for use in landing in place of the runway beacon method outlined in the foregoing description and has been tried with considerable success by personnel of the U.S. Air Corps at Wright Field, Dayton, Ohio. An advantage is that the direction finder indicates the directional heading of the aircraft at all times, thereby obviating such difficulties as "hunting" about the runway beacon course. Other factors of use counterbalance this advantage, however, as will appear from the following. The aircraft is oriented along the proper runway direction by means of direction finder bearings on two fixed ground stations lined up with the runway, and the signals from one of these stations (the one nearer the airport), utilized for direction indication. Wind drift is detected when check bearings on the other ground station show that the aircraft is off course. Correction is made by changing the orientation of the direction-finder loop antenna and, after following the course indications of the first station for an appreciable period, taking a check bearing on the second station. That orientation of the loop antenna is chosen which gives "on-course" readings for the second station. The angle between the new position of the loop antenna and its original position (perpendicular to the longitudinal axis of the aircraft) is equal to the drift angle. For the new loop antenna position, indications from either ground station give the true runway direction provided, of course, that the wind direction and velocity remains unchanged. The bearing of the airplane compass is now noted. Once the glide is started for descent of the aircraft to the landing surface, it is not feasible to keep taking check bearings on the second ground station to determine if wind drift due to changes in wind direction or velocity is taking place. Close check of the airplane compass bearing is required for this purpose, wind drift being observed by a departure of the compass bearing from the value noted. Two instruments are therefore utilized, as in the case of the use of the runway beacon, while a somewhat more involved procedure for determining the true runway direction is required. In actual use, at Wright Field, of this method of runway direction indication, considerable simplification of the above procedure for determining the true runway direction has been found feasible. This is made possible by suitable location of the ground station nearer the airport, the distance of this station from the airport boundary being the important factor. The direction-finder bearings on this station guide the aircraft directly over it, even if wind drift correction is not properly made. Upon arriving over the station, the pilot swings the aircraft so that the compass bearing corresponds to the proper runway direction. If the distance from the airport is not too great, this is sufficient for landing on the runway in spite of cross winds. With this simplification, the use of the direction-finder method becomes comparable with the runway-beacon method for determining the runway direction. Both methods require checking the compass bearing against the radio course indicator. The choice appears to be based on what additional receiving equipment is required on the aircraft. Since practically all commercial transport airplanes carry beacon-receiving equipment, the runway-beacon method is preferable for transport use.

The runway course as defined by either the runway beacon or a direction finder may depart considerably from a straight line due to the presence of long conductors such as telephone and power lines and railroad tracks. The photographic strip of the terrain at College Park shown in figure 5 illustrates this effect. The runway localizing beacon is located at (*a*), and a course oriented in the direction indicated by the three radiating black lines. The true course as determined by numerous flights over a period of many months is actually as shown by the white line. The general bow in the course toward the railroad between points (*b*) and (*c*) may be attributed to the effect of the railroad tracks and telephone lines running parallel to the tracks. The bend in the reverse direction, at (*d*), is apparently caused by the power lines running transversely to the course. It is interesting to note that the bend (*c*), because of its proximity to the airport, gave considerable difficulty during hooded landing tests by causing exaggerated "hunting" about the course just prior to the final landing maneuvers. The adoption of the directional receiving antenna produced a tremendous reduction in troubles from this effect.

The sensitivity of the vertical pointer motion of the combined instrument has been made dependent on the sharpness of course required. The vertical movement of the instrument shown in figure 1 (a) is adjusted to give half-scale deflection on either side of zero for a third of the current required for full-scale deflection. A high degree of sensitivity and attendant course sharpness is thereby maintained about the "on-course" position, while for large deviations "off-course" the sensitivity decreases to permit keeping the deflection within the range of the instrument. The latter feature is valuable when maneuvering about the beacon. Sufficient damping of the instrument pointers is obtained by the use of cobalt chrome magnets, thereby preventing undesired movements due to vibration and extraneous electrical impulses. This minimizes the chances of erratic indications which may confuse the pilot. In early instruments, electrolytic condensers of high capacitance were shunted across the movements operating the pointers to provide the necessary damping.

2. LANDING BEAM

The advantages of the landing beam, foreseen during its early development, are fully realized in its practical application at an airport. The landing path may be so directed that a landing aircraft following the glide path is automatically kept above obstructions without requiring exact knowledge by the pilot of the terrain over which he is passing. Secondly, the average landing path may be made to suit the particular airport merely by adjusting the power of the landing beam transmitter. Each individual aircraft may follow a path departing considerably from the average landing path and more closely suiting its flying characteristics simply by an adjustment of the sensitivity of the landing beam receiving set. The power adjustment at a given airport to secure a suitable average landing path and the sensitivity adjustment on each aircraft to depart by a desired amount from the average path, once made, are permanently fixed. A given aircraft then follows an optimum landing path at all airports equipped with landing beams. As a third advantage, the shape of the landing path is such that the aircraft maintains safe flying speed in following the landing path indications up to the point

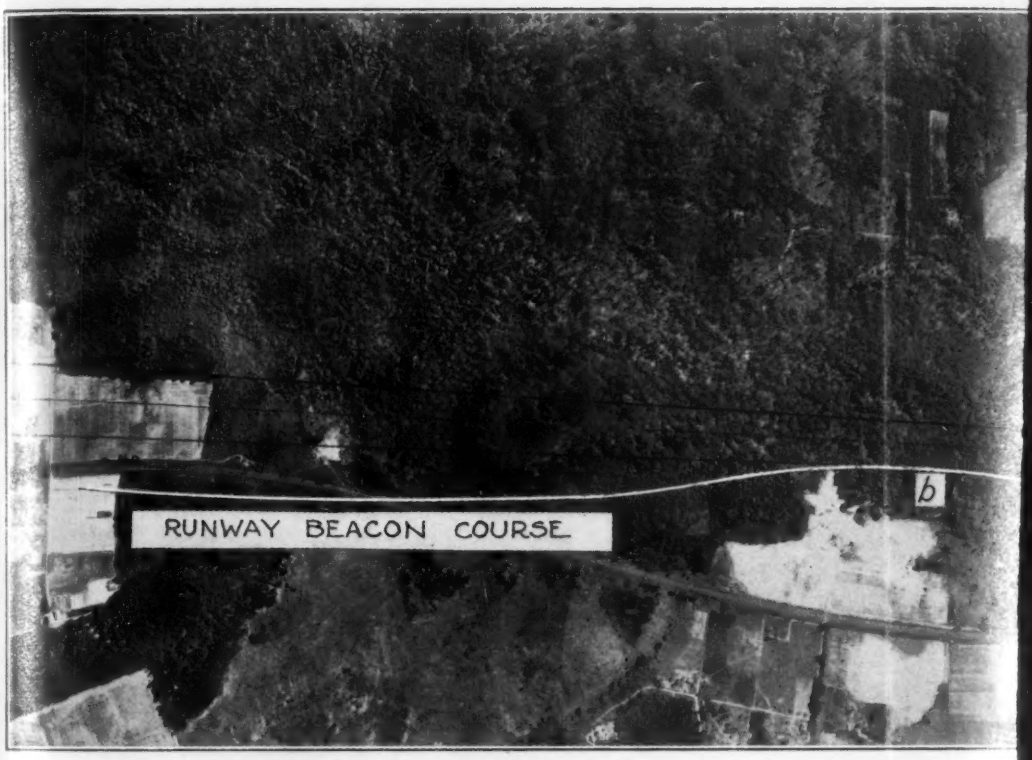


FIGURE 5.—

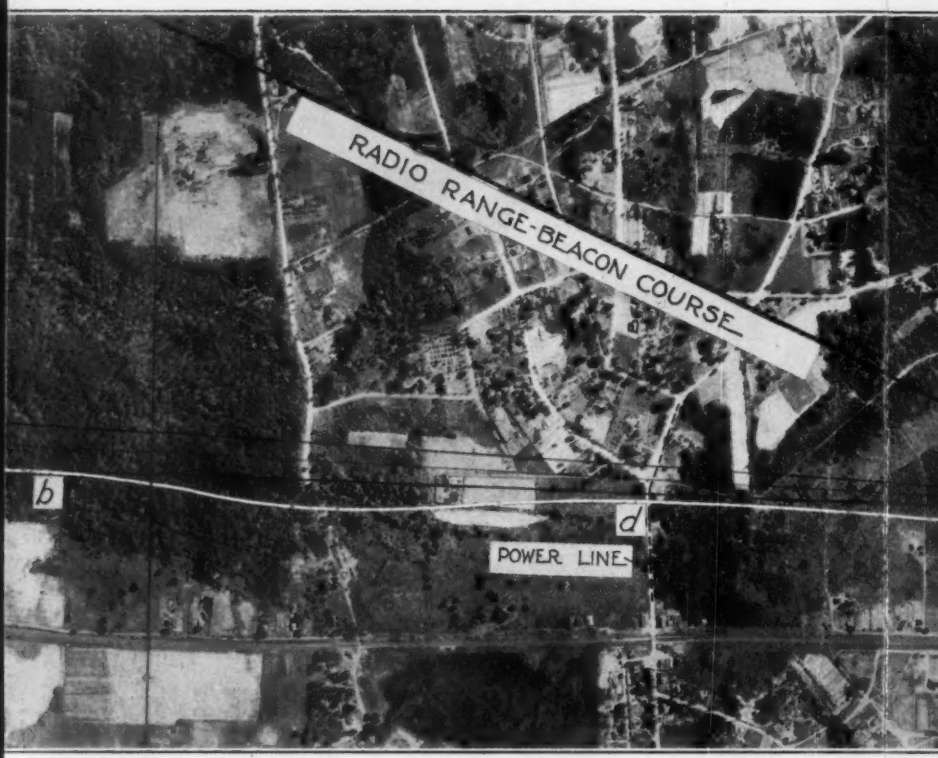
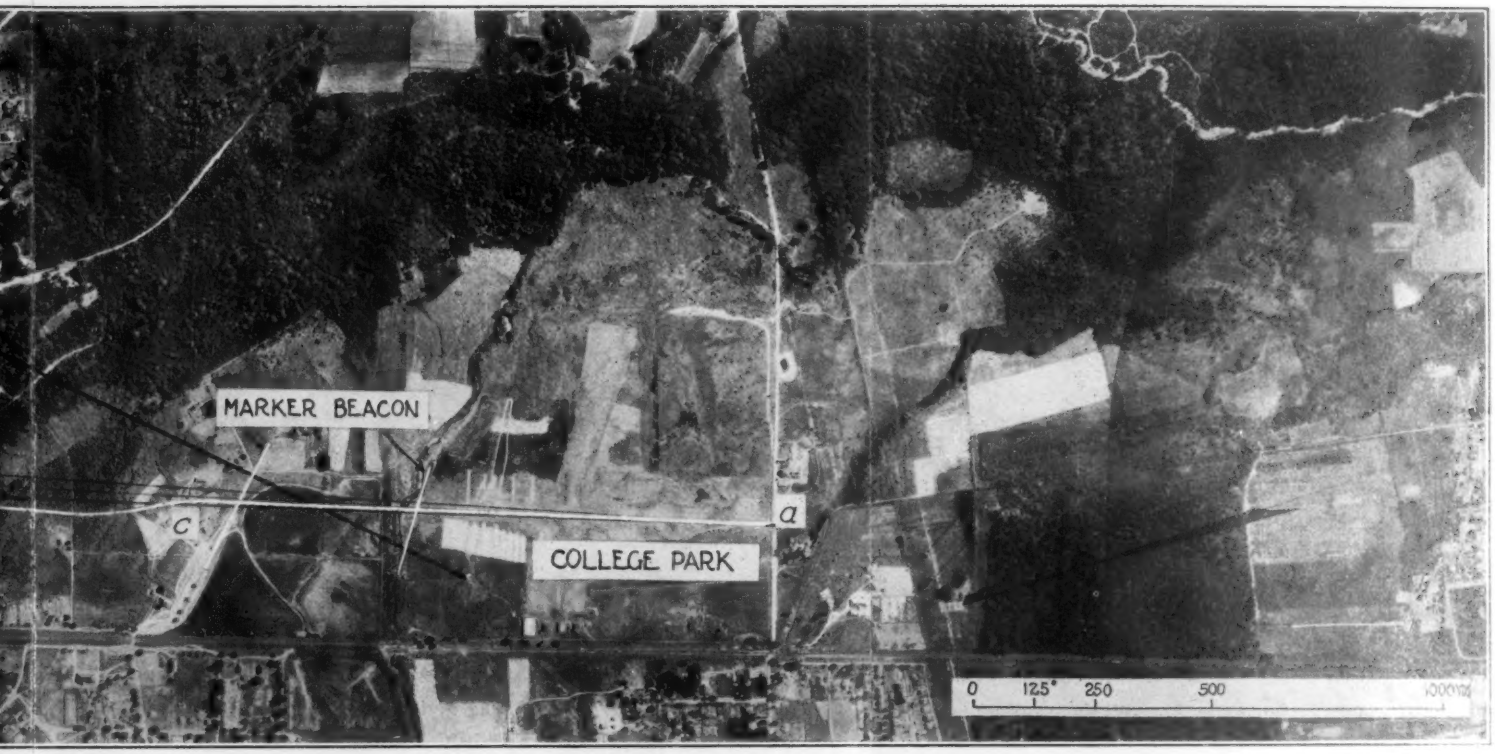


FIGURE 5.—*Photographic strip of College Park, Md., airport showing effect of railroad tracks*



ad tracks and power lines upon shape of runway beacon course.

of receiving the boundary marker beacon signals. The aircraft engine is thus kept at safe operating speed during all maneuvers outside the airport boundaries. A fourth advantage is that the landing glide may be begun at any desired altitude within a rather wide range (say, 500 to 5,000 feet). Beginning the use of the landing beam does not, therefore, involve accurate location of any point at specific distance from the airport, but comes automatically so long as the pilot is following the runway beacon course in the correct direction and at an altitude within the prescribed limits. The fifth advantage

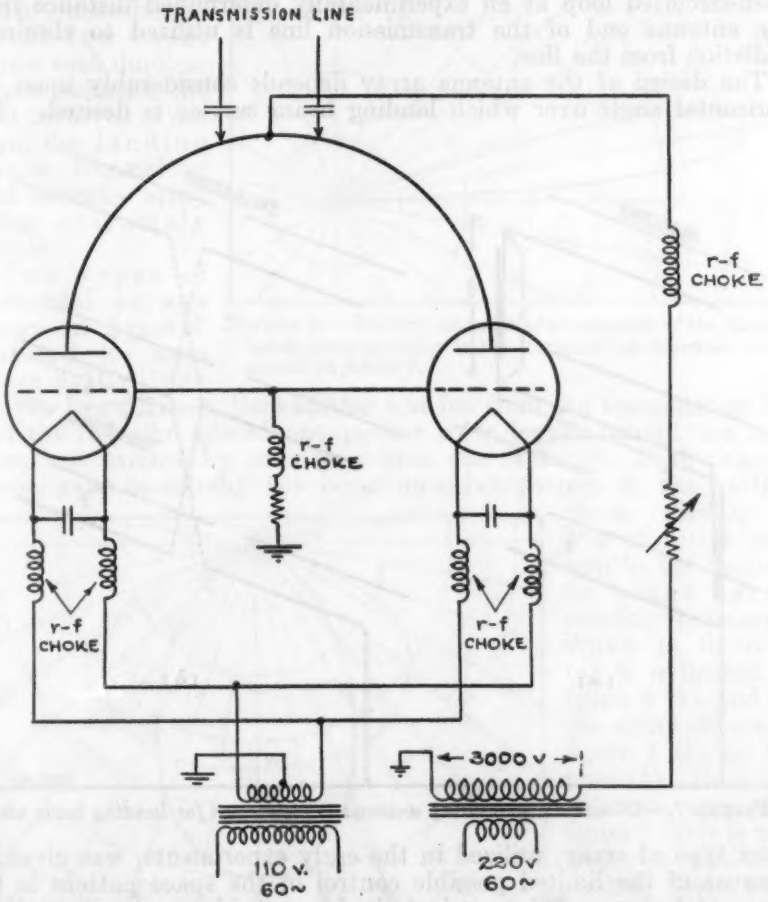


FIGURE 6.—*Electrical circuit arrangement of landing beam oscillator.*

lies in the ease of using the landing beam indications. No manipulation of equipment on the part of the pilot is required. The tuning is fixed. Since a line of constant field intensity is followed no control of volume is necessary. Other advantages will be evident from the following paragraphs.

The landing beam transmitting set may employ any transmitting circuit arrangement which can supply approximately 500 watts to the directive antenna array. Sixty-cycle modulation of the radio-

frequency carrier is required for purposes of audio amplification and filtering in the landing-beam receiving set carried on the aircraft. The oscillatory circuit shown in figure 6, employing two 500-watt 3-element tubes in push-pull, was used at College Park and at Newark. In recent experiments, substituting a quarter-wave transmission line for the filament chokes resulted in considerable increase in the efficiency of the oscillator.

The transmitter feeds power to the directive antenna array by means of a parallel-wire transmission line. A short-circuited or open-circuited loop at an experimentally determined distance from the antenna end of the transmission line is utilized to eliminate radiation from the line.

The design of the antenna array depends considerably upon the horizontal angle over which landing beam service is desired. The

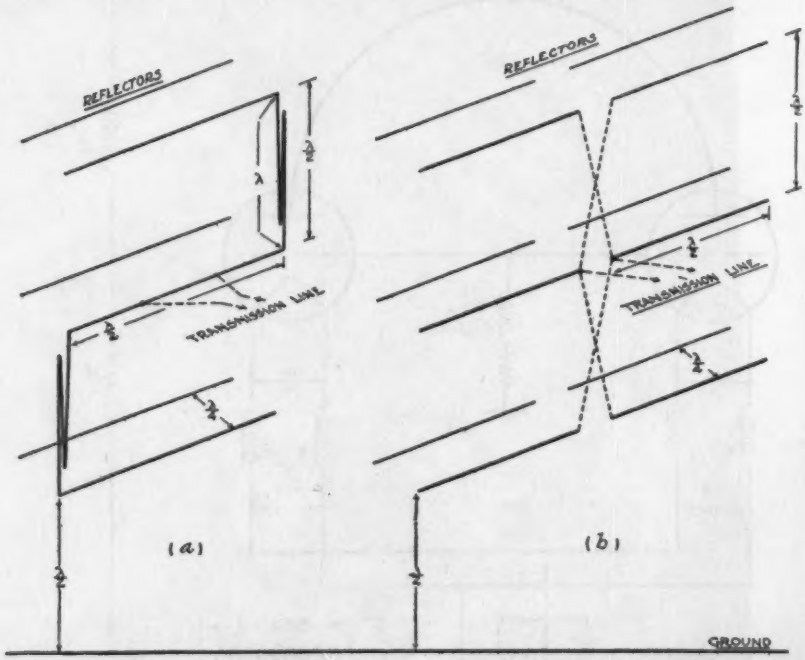


FIGURE 7.—*Directive transmitting antenna arrays tested for landing beam use.*

Yagi type of array, utilized in the early experiments, was given up because of the limited possible control of the space pattern in the horizontal plane. Where it is desired to provide service in all directions, a very simple antenna arrangement, suggested by the Deutsche Versuchsanstalt für Luftfahrt, may be used. This comprises simply a quarterwave vertical antenna, grounded at one end, placed in the center of the airport. The portion of the space pattern used is shaped like a bowl, suggesting the German name, "teller" antenna. With this arrangement, the landing beam transmitter may be placed in a pit in the center of the field. The practical difficulties consist firstly, of the objection to even a low antenna (2.7 feet for 3.3 meters wave length) placed in the center of an airport, and, secondly, the dependence upon the dielectric constant of the ground of the shape of the

space pattern produced with vertically polarized waves. With horizontally polarized waves, such dependence is inappreciable, thereby insuring a fixed landing path under all weather conditions. The above considerations point to the use of a horizontal type antenna array located off the airport, with duplication of the transmitter and antenna array if more than one landing direction is to be served. However, as indicated in section III (1) it is not expected that many airports will require more than a 1-direction installation. In the few cases where such duplication is essential, the cost involved is not high, the landing beam transmitter and antenna array being extremely simple.

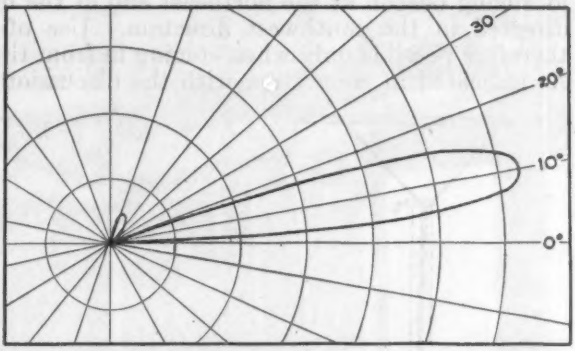


FIGURE 8.—*Vertical directive characteristic of the landing beam corresponding to the transmitting antenna arrays shown in figure 7.*

Two types of horizontal arrays shown in figure 7 (*a*) and (*b*) were tested at College Park. In each case, the radiator was fed from the transmission line and the reflector, placed one-quarter wave length behind the radiator, was excited by induction from the radiator. Both antenna arrays gave essentially the same directive pattern in the vertical plane. (See fig. 8.) The directive pattern in the horizontal plane corresponding to the array shown in figure 7 (*a*) is indicated in figure 9 (*a*), and for the array shown in figure 7 (*b*), by figure 9 (*b*). Using the antenna array of figure 7 (*a*) it is possible to provide landing beam service over a sector of approximately 110°, while with the antenna array of figure 7 (*b*) the useful sector is reduced to approximately 40°. At the limits of these sectors, the landing path for fixed receiving set sensitivity is about 10 percent higher than at the center. This difference is not important in practice. The choice between the two types of antenna arrays lies in consideration of the width of the sector in which service is desired and of the intensity of radiation in the area served for a given transmitter power.

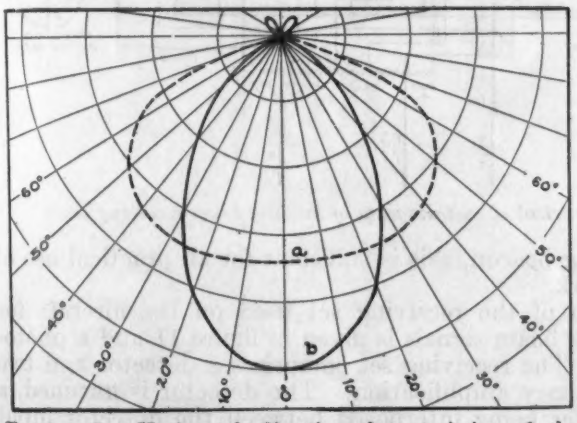


FIGURE 9.—*Horizontal directive characteristics of the landing beam corresponding to the transmitting antenna arrays shown in figure 7.*

At the Newark airport (see fig. 3) the angle between the two hangar lines is approximately 40°. The antenna array chosen is accordingly that shown in figure 7 (*b*). A photograph of this antenna array, as installed at the Newark airport, together with the transmission line feeding it, is given in figure 10. The short-circuited loop for impedance matching is also shown in this photograph. The transmitter and antenna array at Newark are located along side of the runway localizing beacon at the northeast end of the field, and the radiation directed in the southwest direction. Use of the landing beam is therefore possible only when coming in from the southwest quadrant. As indicated in connection with the discussion relative to location of

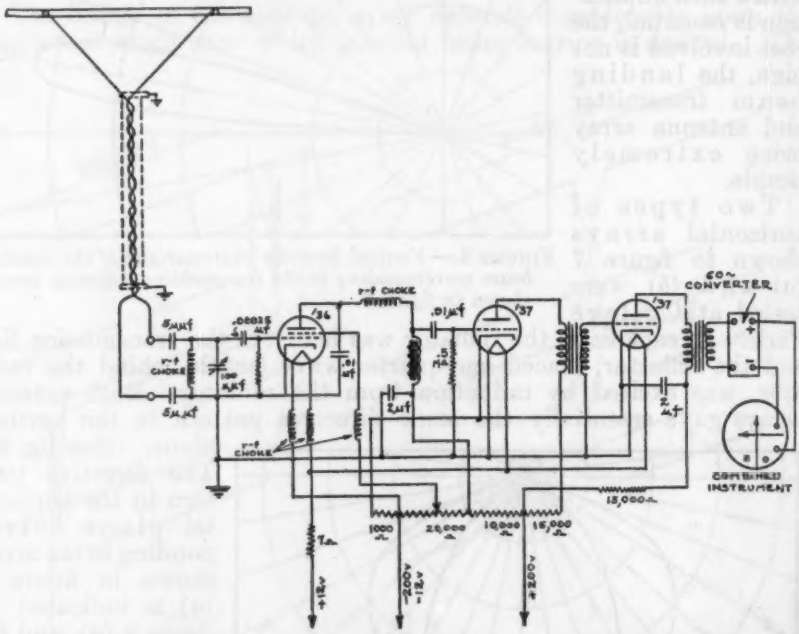


FIGURE 11.—*Electrical circuit diagram of landing beam receiving set.*

the runway localizing beacon, this is sufficient for all practical use of the system at Newark.

A circuit diagram of the receiving set used on the aircraft for receiving the landing beam signals is given in figure 11 and a photograph in figure 12. The receiving set comprises a detector and two stages of audiofrequency amplification. The detector is untuned, a simple high-pass filter being interposed between the detector input circuit and the transmission line feeding it in order to minimize interference from services operating on lower frequencies. The output from the receiving set passes through a mechanical filter, tuned to the modulation frequency at the transmitter (60 cycles) which provides further freedom from interfering signals. Adjustment of the sensitivity of the set is obtained by means of a variable condenser shunting the input to the detector grid. With this condenser the sensitivity of the set may be varied from 10,000 to 40,000 microvolts input for .6 milliwatts output.[4] The sensitivity used in the tests at Newark

[4] These figures are interpolated from measurements made at 10,000 kilocycles.

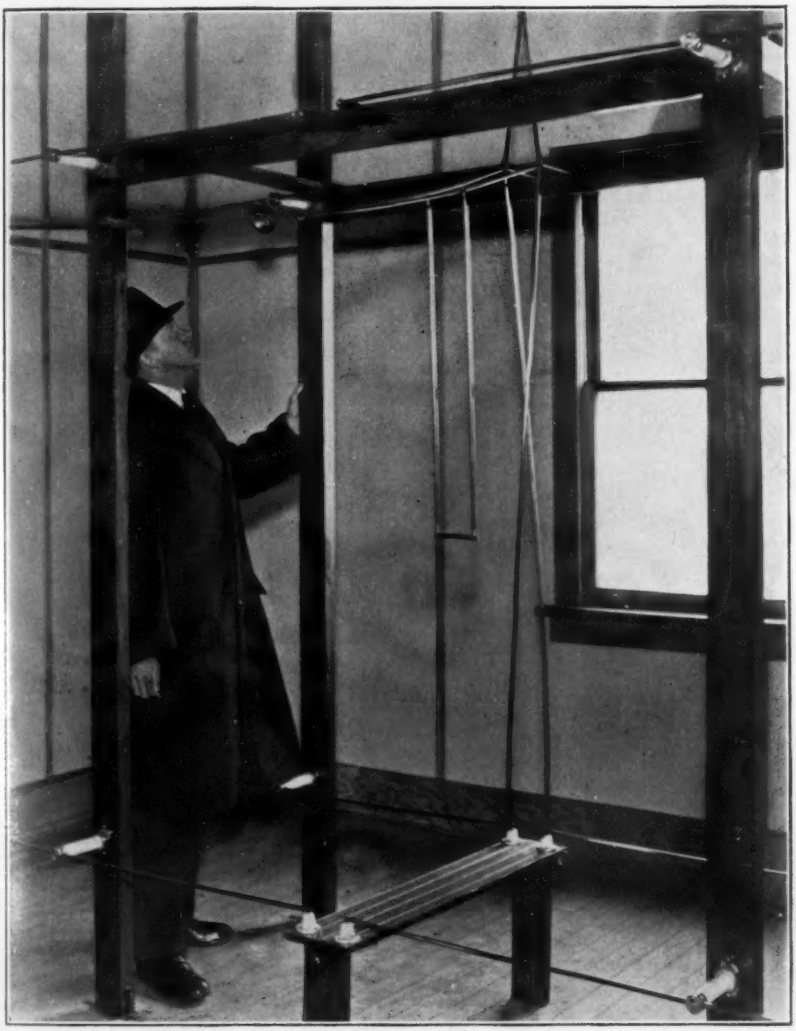


FIGURE 10.—*Landing beam transmitting antenna array employed at the Newark Airport.*

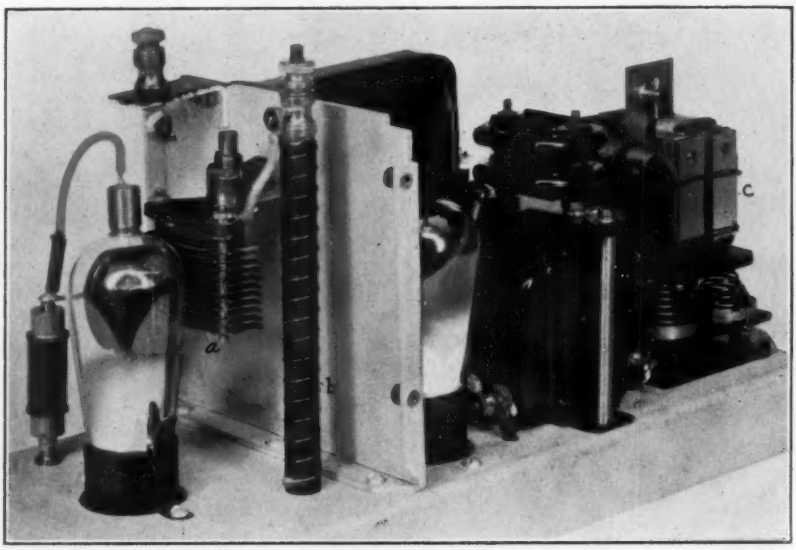


FIGURE 12.—*Photograph showing inside view of landing beam receiving set.*

a, Condenser for permanent adjustment of receiver sensitivity; *b*, portion of high-pass input filter; *c*, mechanical filter tuned to 60 cycles.

and College Park was of the order of 20,000 microvolts. Because of the low receiver sensitivity, the simple means described above for securing freedom from interference have proved entirely satisfactory.

Special attention was given in the design of the receiving set to provide constant sensitivity for wide fluctuations in the supply voltages. Such design is required since the voltage of the storage battery, which serves as the central source of electrical power on the aircraft, may vary considerably even during a single flight. The means adopted are quite simple. Automobile-type receiving tubes are used which are especially designed to work on a wide range of filament voltages; a series resistor is used to step down the 12 volts from the storage battery to the required average value (6 volts). The high voltage supply, furnished by a dynamotor available in the aircraft for use with other receiving equipment, is impressed upon a voltage divider. Taps from the voltage divider provide the proper

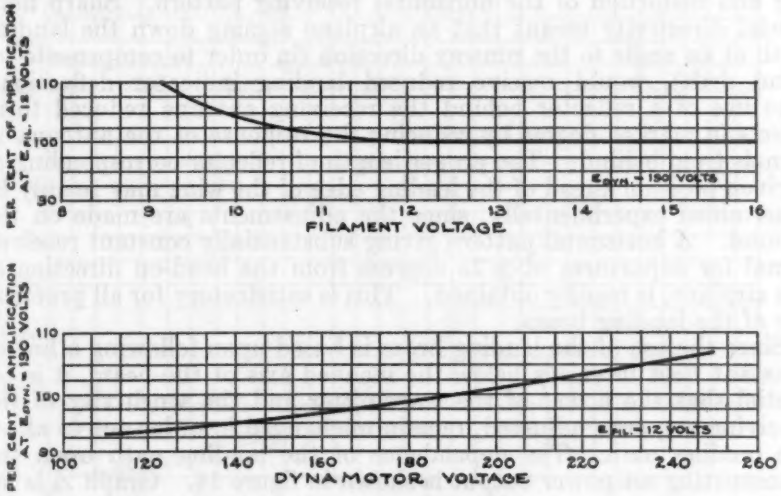


FIGURE 13.—*Effect of variation of supply voltages upon sensitivity of landing beam receiving set.*

voltages to the plate and shield grid of the detector tube and to the plates of the amplifying tubes. These voltages are fixed by test at values giving minimum change of amplification for large departures on either side of these values. Grid detection is used so that variations in the plate and shield grid voltages of the detector tube have minimum effects. Graphs showing the relation between the receiving set sensitivity and the supply voltages are given in figure 13.

The transmission line used for transferring voltage from the receiving antenna to the receiving set consists of a twisted pair of insulated wires inclosed in a metallic shield. Both ends of the shield are grounded to the airplane metallic structure and the transmission line run so that the shield is either insulated from contact with metal parts of the airplane or grounded thereto. The connection of the transmission line to the antenna is indicated in figure 11. The antenna consists of a horizontal copper rod of approximately one half wave length. A reflector is employed to reduce alteration of the space characteristics of the receiving antenna due to its proximity to the aircraft structure. Two locations for the receiving antenna were

tried, one above the wing at the center, and the other ahead of the leading edge of the wing near the wing tip. In the first arrangement, the effect of reflection of the oncoming wave from the body of the airplane was to sharpen up the directivity of reception in the vertical plane, the direction of maximum reception corresponding to an angle above the wing surface. As a result, the deflection of the landing path indicator would increase considerably when the airplane was put into a dive and decrease during a climb. The use of a reflector placed above and somewhat behind the antenna compensated the effect of the airplane, reducing to considerable extent the vertical directivity of reception. However, the length and position of the reflector had to be determined by a long series of flight tests. In the second arrangement, the vertical receiving characteristic was found satisfactory. In the absence of the reflector, however, reflection of the oncoming wave from the leading edge of the wing caused a sharpening and distortion of the horizontal receiving pattern. Sharp horizontal directivity meant that an airplane coming down the landing path at an angle to the runway direction (in order to compensate for wind drift), would receive reduced landing indicator deflections. The use of a reflector behind the receiving antenna reduced these effects in marked degree by reducing the response of the antenna to signals from behind. The proper length of reflector corresponding to a given position ahead of the leading edge of the wing may readily be determined experimentally, since the adjustments are made on the ground. A horizontal pattern giving substantially constant received signal for departures of ± 25 degrees from the head-on direction of the airplane, is readily obtained. This is satisfactory for all practical use of the landing beam.

Since the use of the landing beam is based upon following a line of constant field intensity below the inclined axis of the beam, it is essential that the power of the transmitter and the sensitivity of the receiving set, once adjusted, remain unchanged in order not to affect the landing path. The dependence of the landing path upon the transmitting set power output is shown in figure 14. Graph *A* is for normal power output at Newark, approximately 350 watts. Graph *B* corresponds to 10 percent increase while graph *C* corresponds to 10 percent reduction in power output. These graphs were calculated from the expression given in equation (1) and derived by G. L. Davies of the Bureau of Standards. Experimental checks in an airplane gave excellent agreement with the calculated data. From a study of figure 14 it is evident that the change in the landing path is negligible even for a 10 percent change in power output. Such variation in power is hardly to be expected in practice.

$$x^2 = \frac{8\pi AnIh}{E\lambda}\, y = py \qquad (1)$$

where

x = horizontal distance of airplane from landing beam transmitter
y = height of airplane
A = numerical constant (depending upon field intensity due to single antenna)
n = number of pairs of antennas and reflectors
I = current in each antenna
h = height of center of transmitting array above ground

E = field intensity necessary at receiving antenna to produce on-course indication
λ = wave length

This holds provided $\frac{y}{x} \leqslant 0.05$.

The change in landing path corresponding to ±10 percent change in amplification of the landing beam receiving set is about the same as that indicated in figure 14 for ±10 percent change in power. This is based on the use of square-law detection. A detector of a higher order than square-law would reduce the change in path from that indicated. The desire to minimize the possibility of such change led

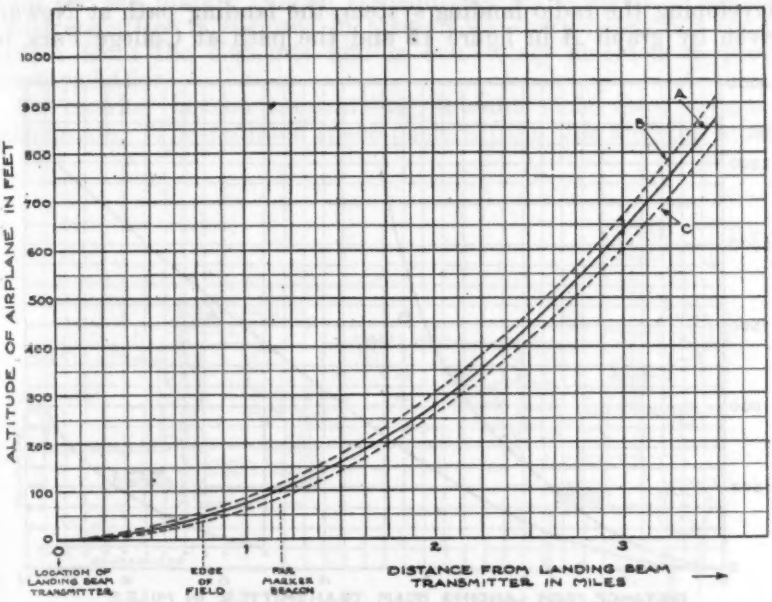


FIGURE 14.—*Effect of changes in transmitting power upon landing path.*

to the use of the simple type of landing beam receiver described. Since there is no tuning nor regeneration, and the sensitivity is not at all critical with variation in supply voltage, the only possibility for change in sensitivity is loss of emission in the receiving tubes. Repeated checks on the two test airplanes for a period of nearly a year showed such possibility to be rather remote. Routine service checks of the equipment at regular intervals are sufficient to eliminate any possibility of improper operation from this source. An effective overall check of the receiving equipment consists of applying a known voltage (of the landing beam frequency) to the landing beam receiving antenna and checking the reading of the landing path indicator. The voltage required to give "on-course" indication, as previously determined and recorded, is a good value to use for this test.

It is of interest at this point to indicate the adjustment procedure to be followed in installing the landing beam at a number of airports. For the purpose of explanation, assume that an installation has been made at one airport, say at Newark, and that it is desired to provide

landing-beam service at a second airport of different characteristics, say at College Park. (The distance of the approach end of the field from the landing-beam transmitter is approximately 4,000 feet at Newark and 2,000 feet at College Park.) Assume furthermore that the receiving sets on a number of airplanes have been adjusted to give suitable landing paths at Newark and are to be used in landings at both airports. The problem is to adjust the power of the landing-beam transmitter at College Park to give satisfactory landing paths corresponding to the receiver sensitivities previously found desirable at Newark. The procedure is simply to calculate the required power to give a suitable average landing path (as determined from obstructions in the path, point of contact with the field, etc.), and to check this experimentally in an airplane. For the two test airplanes used in developing the radio landing system, the landing path at Newark is given by graph *A* in figure 15 and the path at College Park by

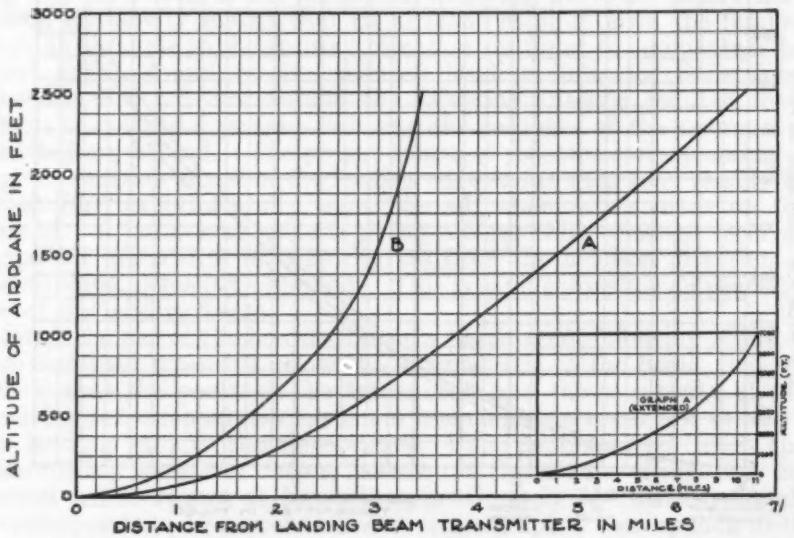


FIGURE 15.—*Landing paths used at Newark and at College Park.*

graph *B*. The antenna power corresponding to graph *A* is approximately 350 watts, and to graph *B*, approximately 175 watts.

Each of the landing paths shown in figure 15 really represents the path of the landing beam receiving antenna. The actual wheel height of a landing airplane is below this path by an amount depending upon the location of the receiving antenna. The difference is relatively unimportant except in determining the point of contact of the wheels with the ground. At Newark, the point of contact is 2,450 feet from the landing beam transmitter for an antenna height of 15 feet, 2,000 feet for an antenna height of 10 feet, and 1,400 feet for an antenna height of only 5 feet. If the antenna is to be considered mounted ahead of the leading edge of the wing, the first case corresponds to the average transport passenger airplane, the second to the average itinerant and mail airplane, and the third to the low-wing monoplane used in mail service. In the first two cases it is feasible to use the landing beam down to the point of contact with the ground.

In the latter case, it is desirable to start settling down for a landing as soon as the boundary marker beacon signal is heard, in order that the airplane will not land too far downfield with the possibility of overrunning the field during taxying.

3. MARKER BEACONS

The function of the marker beacons in the radio landing system was outlined under section II. The transmitting set employed for the marker beacons is very simple, comprising a low-power radio-frequency oscillator, an audio-frequency oscillator providing grid modulation of the radio-frequency oscillator, and a rectifier to permit operation from the 60-cycle supply. The electrical circuit diagram for the marker beacon transmitting set is given in figure 16. The transmitter is enclosed in a small weatherproofed box for protection from the elements and is completely shielded to prevent direct radiation.

The marker beacon transmitting antenna must meet several requirements. The radiated space pattern from this antenna must be

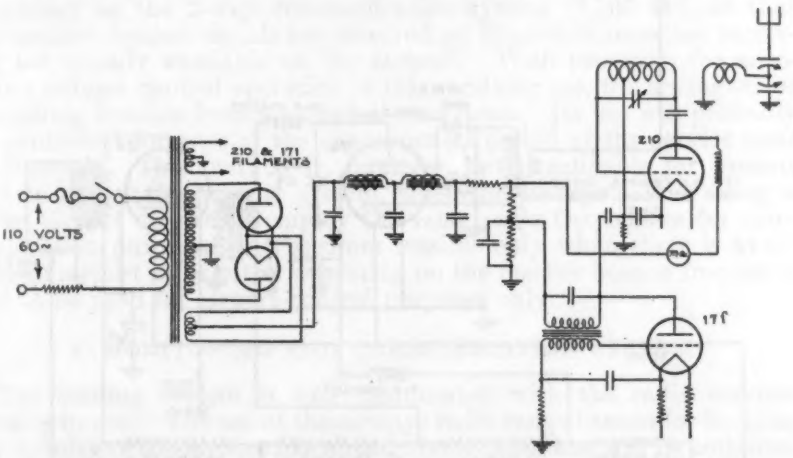


FIGURE 16.—*Electrical circuit diagram of marker beacon transmitting set.*

sharply directive upward forming what is virtually a wall of radio signals through which the aircraft passes. In addition, it must be possible to control the distance along the line of flight of the aircraft over which the signal is heard, thereby securing any desired sharpness of definition of a given boundary line. It must also be possible to provide marker beacon service for any desired distance along the boundary to be defined. The antenna adopted, a long horizontal wire, 2 to 6 feet high and stretched transversely along the line of flight of the aircraft, satisfies the above conditions. For a given receiving set sensitivity, sharpness of definition is controlled by the amount of power fed to the antenna. Service may be given over any length of the boundary line by increasing the length of the antenna. Thus at Newark, to provide service for any orientation of the runway beacon course within the 40° sector, the field marker beacon antenna was made 2,500 feet long and the approach marker beacon antenna 3,500 feet long. Actually the antennas could have been made con-

siderably shorter, since considerable radiation is had off the ends of this type of antenna which may be used as much as a quarter mile from the ends. To eliminate reflection from the ends and consequent setting up of nodes at intervals along the antennas, the antennas are terminated at each end into 600-ohm resistors. Practically uniform service is had when passing over any point of the antennas.

The marker beacon transmitter and transmitting antenna arrangement provide great flexibility relative to the radio frequency on which

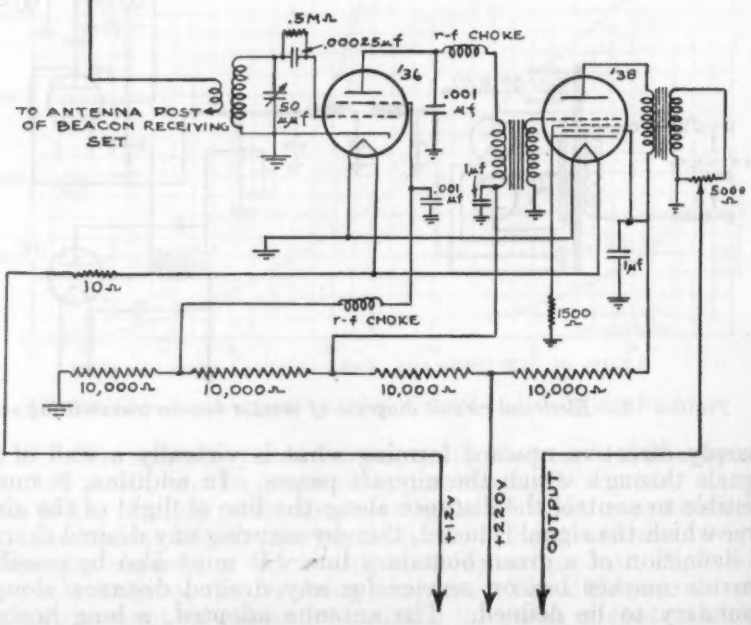


FIGURE 17.—*Electrical circuit diagram of marker beacon receiving set for operation on 10,000 kilocycles.*

they operate. The frequency may be anything from 200 to 20,000 kilocycles without imposing special features of design and is determined solely from considerations of the system as a whole. For application of the system as outlined in the foregoing description, the preferred frequency of operation is of the order of 10,000 kilocycles. A special 2-tube receiving set is then required on the airplane for receiving the marker beacon signals. A circuit diagram of a suitable

receiving set is given in figure 17. The set may be housed in the same chassis as the landing-beam receiving set and fed by the same power supply cable. The antenna coupling arrangement is such that the regular beacon receiving antenna also serves as the marker beacon antenna. The tuning and sensitivity of the marker beacon set once adjusted, require no further attention except for service checks in maintenance of the equipment.

In a series of tests at College Park the marker beacons were operated on the same radio frequency as the runway localizing beacon. The need for a special marker beacon receiving set was thereby obviated, the marker beacon signals being received on the regular beacon receiving set. Close control of the marker beacon power output was, however, required to prevent overloading of the beacon receiving set when the airplane was directly over the marker beacon antennas, since such overloading affected the accuracy of the runway beacon course indications. The tests demonstrated that this method of operation would not prove practicable in service.

A third scheme of operation was tried in the experiments at Newark. In this arrangement the marker beacons operate on the same radio frequency as the 2-way communication system (3,105 kc), so that the marker beacon signals are received on the communication receiving set already available on the aircraft. With provision for automatic volume control operation of this receiving set, the arrangement is entirely feasible from a technical viewpoint. Its use will probably be continued for part of the experimental period of the service tests at Newark. This method is, however, not practicable for present service use of the landing system, since the transport lines using a given airport do not all employ the same radio frequencies for communication purposes. It becomes feasible only when there is available an airport transmitter operating on the marker beacon frequency and to be used for airport-control purposes only.

4. MONITORING AND COMMUNICATION SYSTEM

The landing system is well coordinated with the radio services already in use. The use of the airways radio range beacon for locating the vicinity of the airport has already been noted and will be amplified later on in this paper. The effective use of the 2-way communication system will be apparent from a description of the set-up used at Newark.

In addition to the ground equipment already described, there is installed at the Newark airport, in the National Guard Administration Building, a monitoring panel and a 2-way communication system. The monitoring panel comprises radio-receiving equipment tuned to the runway localizing beacon, marker beacons, and landing beam. Visual indicators show at a glance the status of operation of each transmitter. The 2-way communication system consists of a standard aircraft 50-watt transmitter, operating on 3,105 kilocycles, and a suitable receiving set. With this equipment the operator may keep in constant communication with the pilot of a landing aircraft. Assurance can be given the pilot that the equipment is functioning properly (as seen from the monitoring panel), and that the airport is clear for a landing. While the control room in which this equipment is located overlooks the airport, nevertheless, under conditions of practically zero visibility, it has proved desirable to station a man

at the southwest end of the field to assist in determining that the airport is clear. Remote-control junction boxes are accordingly located at this end of the field, which provide direct 2-way communication with the landing aircraft as well as interphone communication with the control room. The junction boxes and the control cable from the National Guard building are shown in figure 3.

While in the Newark installation the communication service was on the same radio frequency as the marker beacons, the service is in no way affected if the marker beacons are modified to operate on 10,000 kilocycles.

5. COORDINATION OF RECEIVING AND INDICATING EQUIPMENT

Considerable study was given during the tests of the landing system to the problem of minimizing the number of radio controls to be operated by the pilot. In addition to the regular tuning and volume controls already provided on the aircraft for use with the beacon and communication receiving sets, but three switches are required. One of these is the volume-control switch which selects either manual or automatic volume-control operation of the beacon receiving set. The second is the reversing switch for the vertical pointer of the combined instrument, provided so that the deflection of the pointer and the direction of deviation of the aircraft with respect to the course may correspond whether the aircraft is flying away from or toward the runway beacon. The third switch is the "flight-land" switch. In the "flight" position this switch connects the horizontal pointer movement of the combined instrument to the output of the reed converter so as to indicate the volume of received signal in the output of the beacon receiving set. This indication is for the purpose of informing the pilot that his receiving set and the beacon transmitter are functioning properly. Otherwise the vertical pointer, which indicates the beacon course, being of the zero-center type, might read "on-course" with the beacon signal off or the receiving set not functioning. In the "land" position this switch turns on the landing beam and marker beacon receiving sets and connects the horizontal pointer of the combined instrument to function as the landing path indicator, as described in the foregoing.

It is interesting to note that with the "land-flight" switch in the "flight" position, all of the equipment in operation—i.e., the beacon receiving set, the reed converter, the combined instrument, the volume-control switch, and the course-reversing switch—are useful in receiving indications from available visual type airways radio range beacons. The equipment added for landing purposes comprises merely the combined landing beam and marker beacon receiving set.

The coordination of the radio instruments with the flight instruments to provide most convenient use by the pilot constituted an important problem. Typical instrument panel arrangements are shown in figures 18 (*a*) and (*b*). Figure 18 (*a*) is for the panel in the airplane used for the "hooded" landings, while figure 18 (*b*) is for the panel in the cabin airplane used for demonstration purposes. The layout in figure 18 (*a*) is based on the principle of having, as nearly as possible, all the direction indicating instruments in a vertical row, and all the instruments giving attitude of the airplane in a horizontal row. In accordance with this arrangement the combined instrument (because of its vertical pointer), is placed in a

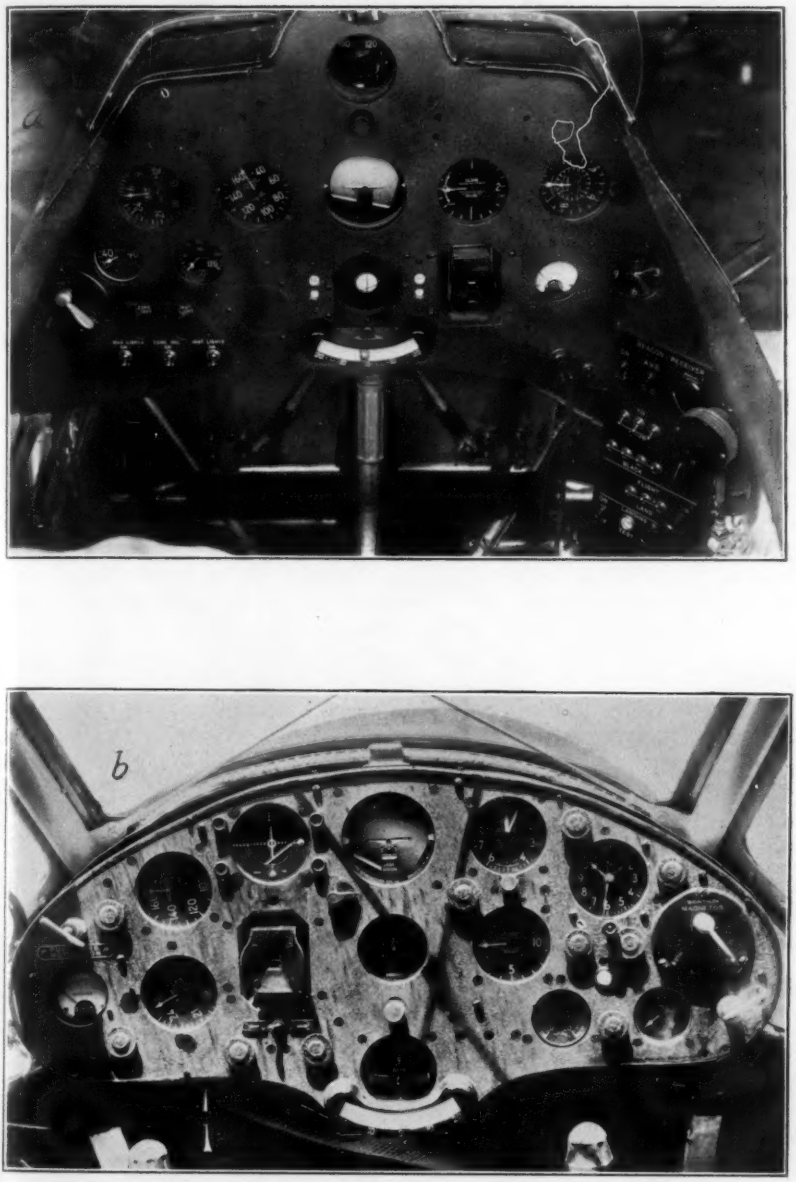


FIGURE 18.—*Typical arrangements of airplane and radio instruments on pilot's instrument panel.*

vertical row with the gyroscopic compass at the top, and the magnetic compass [5] at the bottom—hidden by the control stick. The horizontal row contains, from left to right: The tachometer, the air-speed indicator, the gyroscopic artificial horizon, the rate of climb indicator, and the barometric altimeter. These instruments are mounted so that during normal flight all the pointers are in the horizontal position. Departure of any pointer from this position immediately draws the pilot's attention to the particular instrument giving the off indication. To the right of the combined instrument is seen a reed indicator, and to the right of that, the distance indicator. The location of these instruments is of no particular importance. The reed indicator gives the same indication as the vertical pointer of the combined instrument and was used by the pilots for check purposes only; it is not expected that this instrument will be required in service installations. The distance indicator, being used only occasionally during a flight, may be mounted in any convenient location. Because of limited space, no turn indicator is provided on this instrument panel, the gyroscopic compass serving for this use.

In figure 18 (*b*), the instrument layout is quite different. The idea of placing all the attitude-indicating instruments in a horizontal row is departed from in order that the important instruments may be mounted as close to each other as possible. The need for travel of the pilot's eyes over the instrument panel is thereby reduced. The grouping provides better correlation of the vertical pointer of the combined instrument with the gyroscopic compass, and of the horizontal pointer with the attitude-indicating instruments. The magnetic compass is not shown in this view. It is of interest to note at this point, that different pilots have different preferences for the instrument arrangement on the panel. Nevertheless, the proper grouping of instruments was found to be of utmost importance to the success of the tests on the landing system. It was found, for example, that the demonstration airplane was much easier to fly on the landing system than the airplane used in the hooded tests. This was because of the improved instrument grouping and also because the instrument panel on the demonstration airplane is at a greater distance from the pilot than in the case of the "hooded" airplane, so that more of the instruments are focussed in the pilot's view, thereby providing easier coordination.

IV. OPERATION OF THE SYSTEM

The operation of the radio-landing system may best be explained by outlining several typical examples of its use.

1. METHOD OF MAKING A LANDING DURING FOG

Major use of the system will probably consist of facilitating safe landings during fog conditions. The term "zero ceiling, zero visibility" usually applies to a ceiling height of from 50 to 100 feet and a visibility of from one eighth to one quarter mile. Under these conditions, use of the system is extremely simple, as may be shown by an account of an actual blind flight from College Park, Md., to the Newark Airport, during which radio was the sole means used for navigation. The flight was made on March 20, 1933, as a practical

[5] The magnetic compass is needed only for setting the gyroscopic compass to correct for precession.

demonstration of the landing system. The flight crew consisted of J. L. Kinney, pilot, W. W. La Violette, mechanician, and the writer. The ceiling was so low, upon starting from College Park, that it was found impossible to fly below it. Pilot Kinney therefore maneuvered the aircraft to an altitude of 3,000 feet and maintained this average altitude throughout the rest of the flight. At this altitude no sight of the ground or sky was had except for an instant over Baltimore. The fog was like a milky blanket surrounding the aircraft. Guidance from College Park to Hadley Field, New Brunswick, N.J., was obtained through the use of the Washington and Hadley Field aural type radio range beacons. After passing through the "zero-signal" zone of the Hadley station, the beacon-receiving set was tuned to the frequency of the runway beacon at the Newark Airport (278 kc). The vertical pointer of the combined instrument showed approximately "on-course", the projection of the northeast-southwest runway at Newark being approximately over the Hadley station. The distance indicator was off-scale, showing that the airplane was more than 5 miles distant from the runway-beacon transmitter. The "flight-land" switch was thrown to "land", thereby turning on the landing beam receiving set and connecting the horizontal pointer of the combined instrument for operation from the output of this set.

Flying at 3,000 feet altitude, the horizontal pointer of the combined instrument began to deflect, gradually reaching the horizontal "on-course" position. This corresponded to a distance of 8 miles from the landing-beam transmitter, as determined from the normal landing path at Newark. (See fig. 15, graph *A*.) After communicating with the 2-way communication station at the airport to determine that the field was clear for a landing, the pilot maneuvered the airplane to keep the two pointers intersecting over the small circle in the center of the combined instrument dial. This gradually brought the aircraft along the runway direction and down the landing path shown in figure 15. (See also fig. 3.) Since the wind was from the northeast quadrant and of about 6 miles velocity, no particular effort was required for staying on both courses. The distance indicator showed continuous approach to the airport boundary, and the barometric altimeter (while not sufficiently accurate for landing) showed continuous approach of the aircraft to ground. When within about 2,000 feet from the southwest edge of the airport, the high-pitched marker beacon signal began to be heard becoming most intense at 1,700 feet from the edge and receding beyond that point. At this stage of the landing the ground became visible for the first time, the aircraft being somewhat over 100 feet above the ground. The pilot proceeded to land visually. Had the ground not become visible, the pilot would have continued at the same engine speed until the low-pitched field marker beacon signal was heard. He would then throttle down the engine and pull back the control stick so as to be in correct position for a 3-point landing when contacting the ground.

Several modifications of the above procedure for landing might arise, particularly at the start of the landing maneuvers, depending upon the location of the airways radio range beacon with respect to the airport and also upon the direction of flight. For example, in the case of a flight from Hartford, Conn., to Newark, N.J., the northeast course of the Hadley Field radio range beacon would be followed. Since this course passes within a few miles of the Newark airport, the

pilot would not proceed as far as the "zero-signal" zone over the Hadley Field station, but would tune to the frequency of the runway localizing beacon at Newark, at intervals during the estimated last 30 miles of the flight, until signals from the runway beacon were received. He would then employ the course indications from this beacon in the standard way for locating the airport and orienting the aircraft along the desired landing direction. Again, consider a case where the airways radio range beacon is located, say, northwest of an airport and a pilot coming in from the northwest direction wishes to land due west along the east-west runway. Upon reaching the "zero-signal" zone over the radio range-beacon station, the pilot would tune his set to the frequency of the runway beacon. Knowing the geography of the field, he would circle the runway beacon at fixed radius (as determined from the distance indicator) and would count the number of courses passed through. In this case, the third course would be the one he would follow in order to secure the desired landing direction.

Other departures from the landing procedure outlined occur when the wind direction is not from the quadrant for which landing service is provided. Referring to the Newark airport installation shown in figure 3, assume that the ceiling is 100 to 200 feet with a northwest wind of 15 to 30 miles per hour velocity. In following the spatial landing path, the pilot must now "crab" into the wind in order not to drift east of the runway course. The exact angle of "crab" is determined by trial and error as indicated in section III (1). Upon breaking through the ceiling, the pilot maneuvers the aircraft below the ceiling in order to land into the wind. A very interesting example of such a landing was experienced during demonstrations of the system at Newark on April 6, 1933. Rain and sleet conditions were prevailing with a ceiling of somewhat over 100 feet. When at about 5 miles southwest of the airport, the wind was from the southwest being quite gusty and attaining a velocity of possibly 40 miles per hour. At approximately 3 miles from the airport a wind-shift line was encountered, the wind direction changing suddenly to northwest. In spite of the gustiness, and the sudden change from a strong tail wind to a strong cross wind, the pilot was able to follow accurately the indications on the combined instrument. Upon coming within sight of the ground (at 100 feet), he circled the field at this altitude and landed into the wind.

Consider another case, where the ceiling is 50 feet or less and the wind from the northwest direction, of 10 to 15 miles per hour velocity. In following the runway beacon course, the pilot determines the angle of "crab" to compensate for wind drift and maintains the proper compass bearing to effect proper wind-drift correction. This compass bearing is followed until after hearing the signals from the approach marker beacon. As soon as the field marker beacon is heard, and assuming that the ground is then not in sight, the pilot swings the airplane to assume the true compass bearing of the runway, thereby preventing running off the runway after landing. It is interesting to note that while fog conditions corresponding to this case were not encountered during the tests at Newark, many of the hooded landings to be described in the following were made with cross winds of from 10 to 20 miles. The simulated visibility for these tests was, of course, absolute zero.

2. USE OF SYSTEM IN EVENT OF FAILURE OF ONE OF ITS ELEMENTS

A remarkable feature of the radio landing system is that it is possible to shut off any one of its three elements and still furnish enough information to permit an emergency landing.

Assume first that the two marker beacons are not operating. Warning of close approach to the airport is then given by the indications of the sensitive barometric altimeter (corrected for barometric pressure and for the altitude of the airport). The pilot following the landing beam indications notes that the altimeter shows closer and closer proximity to the ground. Obviously, he must be approaching the airport. This information permits him to reduce his landing speed so that contact with the ground will not occur at too high a speed.

Next assume that the landing beam is shut off. In this case, one end of the field is defined by the marker beacons and the opposite end by the "zero-signal" zone over the runway beacon. The runway direction is, of course, also defined. The procedure for landing consists in finding the "zero-signal" zone over the runway beacon, then following out the runway course in the direction of the marker beacons at, say, an altitude of 1,000 feet. The time taken to reach the marker beacons is then a measure of the ground speed of the airplane. Maintaining the same altitude and the same engine speed, the pilot continues to follow the runway course in the same direction until he computes his position to be, say, 3 miles from the airport. The distance indicator serves as a check on this computation. The pilot then makes a 180° turn and reduces his engine speed to start a glide which, if begun 3 miles from the airport at 1,000 feet altitude, is calculated to land the airplane somewhere near the center of the field. The runway-course indications serve to check wind drift, while the marker-beacon signals operating in conjunction with the sensitive barometric altimeter substitute for the landing beam in giving a measure of the height of the airplane as it crosses the airport boundary.

Finally, assume that the runway localizing beacon is not operating. In this case, the desired landing runway direction is approximately located from a knowledge of the orientation of the runway with respect to the main radio range-beacon station and by use of the compass. Advantage is taken of the directional properties of the landing beam receiving antenna to secure more definite orientation along the true landing direction. As noted under section III (2), the received landing beam signal measured on the horizontal pointer of the combined instrument is a maximum when the nose of the airplane is pointed at the landing beam transmitter and decreases for angular departures of the airplane on either side of this heading. The effect is purposely broadened to provide minimum reduction for $\pm 25°$ change in heading of the aircraft, in order that the pilot does not lose the landing beam indications when "crabbing" due to cross winds. However, close observation permits noticing the effect for deviations of $\pm 5°$. The landing-beam receiver may therefore also be used as a direction finder and, in conjunction with the compass, is capable of determining the true runway direction.

Each of the above three problems were actually worked out in flight tests in a hooded airplane. The possibility of effecting a safe

landing in spite of the failure of any one of the three elements of the system has added materially to the confidence of the test pilots in the practicability of the system.

3. SIMPLIFICATION OF SYSTEM FOR ECONOMY PURPOSE

The foregoing discussion suggests that it may prove feasible to reduce the number of elements of the landing system with a view to reduction in cost. This procedure is not considered desirable. The indications given by the combination of the three elements are so definite and instinctive that breaking up the combination is not to be considered except in emergency cases.

One avenue of economy lies in simplification of the runway localizing beacon, the cost of which is estimated as approximately 50 to 60 percent of the cost of the complete ground installation. One arrangement suggested is to use an aural type runway beacon. Some saving is then obtained in the transmitting equipment and, in addition, the need for the reed converter and combined instrument at the receiving end is obviated. The vertical pointer of the combined instrument is then not required and a simple direct-current micro-ammeter may be used for the landing path indications. The lack of sharpness of the aural runway course indications, the need for manual control of sensitivity of the beacon receiving set as the airplane approaches the transmitter, and the lack of automatic coordination of the landing path and runway course indications, are disadvantages of this arrangement.

A second and preferable arrangement results in material cost reduction without at the same time affecting the service rendered. This consists of so locating the airways radio range beacon that one of its courses coincides with the major landing direction of the airport. Where feasible, the entire cost of the runway beacon is eliminated. If the airways radio range beacon is of the visual type, the service is in no way altered. This arrangement was tested at Newark, using a course oriented along the Newark runway from the simultaneous phone and beacon located at Elizabeth, N.J., and was found entirely practicable. The beacon service given by the Elizabeth station is of the combined visual and aural type described in the June 1, 1933, issue of the Air Commerce Bulletin.

V. PERFORMANCE DATA AND TESTS

1. TESTS AT COLLEGE PARK, MD.

The experimental installation at the Newark airport was preceded by exhaustive tests at College Park, Md., where the practicability of the system was studied by means of flights and landings in an airplane equipped with a canvas hood over the pilot's cockpit (rear cockpit of airplane in fig. 6). The first completely blind landing was made by Pilot M. S. Boggs on September 5, 1931. Over a hundred hooded landings were subsequently made by Mr. Boggs. A check pilot was used in the front cockpit to take care of faulty landings or other emergencies. The remarkable accuracy of the system was well established by these hooded landings. The landing runway at College Park is only 2,000 feet long and 100 feet wide with the approach

unobstructed for a lateral distance of only 300 feet. Off the runway, the landing field is quite rough. In consequence, the runway beacon course had to be made sufficiently sharp to give appreciable off-course indications on the aircraft for deviations of the order of 20 feet (at the approach end of the field). At a distance of 5 miles from the transmitter, a deviation of only 250 feet from the runway course could be detected. The actual shape of the runway course is given in figure 5. Tests over an extended period of time showed the course to be fixed in space regardless of weather conditions.

Obstructions in the approach at College Park also necessitated close control of the landing path. To secure a suitable landing path it was necessary for the airplane to clear a house chimney located approximately 1,500 feet from the edge of the field by about 8 feet. The necessary fixed landing path was obtained by maintaining approximately constant power output at the landing beam transmitter and through the use of the landing beam receiving set described in section III (2). It is interesting to note that from the beginning of use of this receiving set, no adjustments of receiver sensitivity were required over a period of nearly a year, and that but two receiving-tube replacements were made during this period.

Theoretical consideration shows that the landing path indications should increase in sharpness as the aircraft approaches the ground. This was clearly shown in figure 30 of the paper of reference 1. Test data taken at College Park corroborate this conclusion. Departures from the landing path of ± 5 feet could be readily detected when the aircraft was over the edge of the field (at 30 feet altitude) while deviations of ± 50 feet were apparent at a distance of 3 miles from the airport (1,600 feet altitude).

An extended study was made of the possible effect of varying ground conditions due to changes in weather upon the shape of the landing path. The path was found to remain exactly the same under all weather conditions with one exception. After a heavy snowstorm in February 1933 the ground was covered with about 8 inches of dry snow having a hard surface crust. The landing path was then found to be raised as follows from the true path indicated by graph B of figure 15. At 3 miles distant from the landing beam transmitter, the new altitude was 2,000 feet instead of 1,600 feet; at 2 miles, it was 800 feet instead of 650 feet; at 1 mile, it was 205 feet instead of 175 feet; while at the approach end of the field, it became 35 feet instead of 30 feet. The landing path could still be followed down to a landing, although steeper than usual. Flight tests during the following few days showed that the path gradually returned to its normal shape as the snow melted, reaching this normal shape as soon as the snow crust was dissolved and before spots of ground could be seen through the snow. In other snowstorms, the raising of the landing path did not occur, probably because of the absence of the peculiar reflecting conditions from the snow crust obtaining in this particular instance.

2. DEMONSTRATIONS AND TESTS AT NEWARK, N.J.

Upon completion of the tests at College Park, it was decided to make an installation at Newark to determine the operation of the system under the conditions obtaining at a busy commercial airport. Other objects of the installation were to focus attention on the prac-

ticability of the landing system, to cooperate with manufacturers in making the equipment commercially available and to cooperate with air transport operators in experimental use of the system.

The tests and demonstrations at Newark began in February 1933 and have continued to date. Two test airplanes were used, one being equipped with two cockpits for hooded landings and the second, a cabin airplane for demonstration purposes. Besides making a large number of hooded landings, it was possible to fly at all times when the scheduled air mail and passenger airplanes were on the ground because of fog. The operation of the system was demonstrated in the air to many engineers and officials as well as to nearly 100 air-transport pilots. It is interesting to note that not even a minor adjustment of the transmitting equipment nor of the receiving equipment on the two test airplanes was required (other than ordinary maintenance), during the entire period of the tests to date.

A few of the more important tests and demonstrations are summarized herewith.

March 1: Pilot Kinney made three excellent hooded landings. Prevailing high cross winds had limited total previous practice under the hood to 3 hours.

March 2: Flight demonstrations of the system were made with leading press representatives as passengers during a snowstorm with strong cross winds and a ceiling of about 200 feet.

March 7: Pilot Kinney made 12 excellent consecutive hooded landings.

March 14: Numerous demonstration flight tests were made under conditions of very low visibility, 100 to 200 feet ceiling. Engineers and officials who were given the flight demonstrations lost some of the nicety of operation of the system since they could not see their position relative to the ground except during the latter portions of the landings.

March 15: Pilot Kinney made three solo take-offs and landings under conditions of very low ceiling and visibility (approximately 50-foot ceiling and one eighth mile visibility). All other flying to or from the airport was interrupted at this time.

March 20: Completely blind flight made from College Park, Md., to the Newark Airport. Details of this flight are in section IV (1).

March 31: Pilot Kinney made several demonstration hooded take-offs and landings in the presence of moving-picture news-reel photographers and representatives of the press. Col. Clarence M. Young served as check pilot.

April 3, a.m.: A representative of the press with some flying ability was taken up as check pilot and Pilot Kinney made a hooded take-off and landing.

April 3, p.m.: After a thorough inspection of the ground equipment and a preliminary flight in the cabin airplane, Col. Charles A. Lindberg made two hooded landings with but slight assistance from Mr. Kinney, serving as check pilot.

April 4 to 6: Heavy fog conditions occurred on each morning of these dates, which interrupted all flying to or from the Newark Airport. Pilot Kinney made a large number of solo take-offs and landings each morning. On two occasions the fog extended only up to about 2,000 feet altitude, above which the sun could be seen. Still, several transport airplanes had to wait on the ground at airports

within 100 to 200 miles from Newark until they were assured that the fog had dissipated.

April 6: A demonstration of the system was given in the cabin airplane during driving rain and sleet and low visibility. The runway beacon and landing beam course indications were accurately followed even though a wind shift line was encountered during the flight. Details of this flight are given in section IV (1).

The system has proved inherently simple to use by pilots. Experience indicates that a good pilot, already experienced in instrument flying, becomes thoroughly acquainted with the use of the landing system after about 5 hours' hood practice. He is then capable of making consistent hooded landings which approach very nearly the type of landings he would make during good visibility.

The success of the tests and demonstrations has aroused considerable interest on the part of air line pilots, air transport operators, radio and aircraft manufacturers, and air officials of a number of foreign countries. Several radio manufacturers are now actively engaged in the manufacture of ground and aircraft radio equipment for use with this system. Information has been received that a number of the foreign countries are planning experimental installations. It is hoped that service tests of the system in this country will begin in the near future.

VI. ACKNOWLEDGMENTS

In conclusion the author wishes to acknowledge the parts played in this development by his coworkers in the Bureau of Standards and in the Aeronautics Branch. Appreciation is due F. W. Dunmore for basic contributions during the development of the system and to G. L. Davies for research in the design and operation of the landing beam transmitting and receiving equipment.

Special appreciation is due Pilot M. S. Boggs for basic contributions to the practicability of the system during the tests at College Park. These tests were begun when there was still but little use and knowledge of instrument flying. Mr. Boggs' unfailing enthusiasm and keen perception of the practical requirements for instrument landings were responsible for many modifications in the system. The experience in instrument flying gained by Mr. Boggs during the tests at College Park was also largely responsible for the adoption by the Department of Commerce of the Scheduled Air Transport Rating for air line pilots, effective January 1, 1933, which requires a thorough knowledge of instrument flying and of locating a given radio range-beacon station or any one of its courses in hooded flight tests.

Special acknowledgment is also due Pilot J. L. Kinney for valuable contributions and for skillful and courageous testing and demonstrations of the system at Newark. Mr. Boggs was killed while on other air-line duties in the southwest during the period of the installation work at Newark and Mr. Kinney, who served as check pilot during some of the tests at College Park, took up the test work at this point in a most satisfactory manner. Acknowledgment is also due E. Yuravich for aid in serving as check pilot during the Newark tests.

WASHINGTON, August 1, 1933.

U.S. Department of Commerce, Bureau of Standards

RESEARCH PAPER RP603

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

TESTS OF THEATRE-PROSCENIUM CURTAINS

By Nolan D. Mitchell

ABSTRACT

Two types of steel curtains, four asbestos cloth curtains, and several kinds of asbestos cloth were subjected to fire tests. The single-ply wire-reinforced curtain of heavy asbestos cloth and the 2-ply curtain of plain asbestos cloth permitted the passage of smoke and flame during the test and glowed on the unexposed surface. Curtains made of two plies of wire-reinforced asbestos cloth separated by a steel frame afforded protection for about 15 minutes against a severe test fire. The steel curtains were tested for one half hour and gave indication of affording protection for somewhat longer periods. Suggested requirements for inclusion in building codes are available.

CONTENTS

	Page
I. Introduction	492
II. Fire tests of curtains	492
1. Testing equipment and methods of testing	492
2. Curtains tested and results	493
(*a*) Curtain *A*	498
(*b*) Curtain *B*	498
(*c*) Curtain *C*	500
(*d*) Curtain *D*	500
(*e*) Curtain *E*	501
(*f*) Curtain *F*	501
(*g*) Comparative performance	504
III. Operating tests of curtains	505
1. Equipment and methods	505
2. Sliding friction of curtains and guides	505
(*a*) Operating tests of asbestos cloth curtains	505
(*b*) Results of friction tests of metal blocks on guide rails	506
IV. Fire tests with decorative paints	506
1. Testing methods and results	507
(*a*) Bunsen-burner tests	507
(*b*) Panel tests	507
V. Tests of asbestos cloth	507
1. Strength and stretch tests	507
(*a*) Equipment and methods	507
(*b*) Asbestos cloths	508
(*c*) Strength tests at ordinary temperatures	508
(*d*) Strength tests at elevated temperatures	508
(*e*) Strength of cloth from tested curtains	510
(*f*) Stretch of asbestos cloths	510
2. Permeability to air	510
3. Tests for soluble and carbonaceous materials in asbestos cloths	512
VI. Conclusions	512
1. Fire resistance	512
2. Operating mechanism	513
VII. Acknowledgments	513

I. INTRODUCTION

The theatre-proscenium curtain serves to close the opening through which the stage is viewed from the auditorium. Most cities require that such curtains be fire resistive because of occasional disasters resulting in great loss of life from fires on the stage. There is, however, great diversity in the requirements of city building codes with respect to the materials, construction, mounting, and operation of proscenium curtains. Even within the same city such curtains may cover a considerable range in these respects as was disclosed by surveys and inspections of several of the theatre-proscenium curtain installations in three cities. From these inspections the question arose as to what kinds of curtains would give the desired degree of protection to theatre audiences. The answer could be given, at least in part, from the results of tests; accordingly the tests reported herein were made.

Preliminary investigations indicated the desirability of making fire tests of as large sections of typical curtain constructions as feasible. The tests, therefore, were made by exposing one side of the curtains to fire in a large furnace used for fire tests of walls and partitions. The tests included two steel curtains and four asbestos cloth curtains. Effects of the fire exposure were visually observed during the progress of the test, and temperatures on the unexposed surface of the curtain as well as in the furnace were measured. Modifications of the equipment were made to study the behavior of the flexible asbestos cloth curtains when subjected to air pressure or both fire and pressure simultaneously.

II. FIRE TESTS OF CURTAINS

1. TESTING EQUIPMENT AND METHODS OF TESTING

The furnace, which accommodates panels 16 by 11 feet in size, is shown in plan and section in figure 1. Two blower fans produced the pressures within the furnace and the pull required to lower the curtains in the operating tests was measured by means of spring scales attached to the hauling cables. Temperatures were measured with base metal thermocouples, those on the surface of the curtains being covered with asbestos paper.

A schedule of heating was chosen to attain an average indicated furnace temperature of 1,700° F. at 15 minutes as compared with one hour specified by standard testing procedure [1] for most other classes of building construction. The choice was made because of the requirement specified by some cities that one side of such curtains must withstand a temperature of 2,000° F. (1,093° C.) for a given time without occurrence of temperature on the opposite side above some specified point, such as 350° F. (177° C.). The temperature rises were as indicated by figures 2 and 3, and it can be seen that those for the furnace were somewhat less than intended.

Two steel curtains, *A* and *B*, were subjected to fire tests for periods of 30 and 35½ minutes, respectively. The four asbestos cloth curtains were subjected for 15 minutes to fires of the intensity shown by the curves in figure 3. Three of them were also subjected to air pressure while exposed to fire.

[1] Tentative Standard Specification for Fire Tests of Building Construction and Materials, now before the American Standards Association for adoption as American Standard, 1933.

2. CURTAINS TESTED AND RESULTS

The curtain specimens were with one exception as large as the panel test frame of the furnace would accommodate. The sizes and general details of construction are given in table 1. The test specimens representative of steel theatre curtains were made as sections of actual curtain constructions. The asbestos cloth curtains were de-

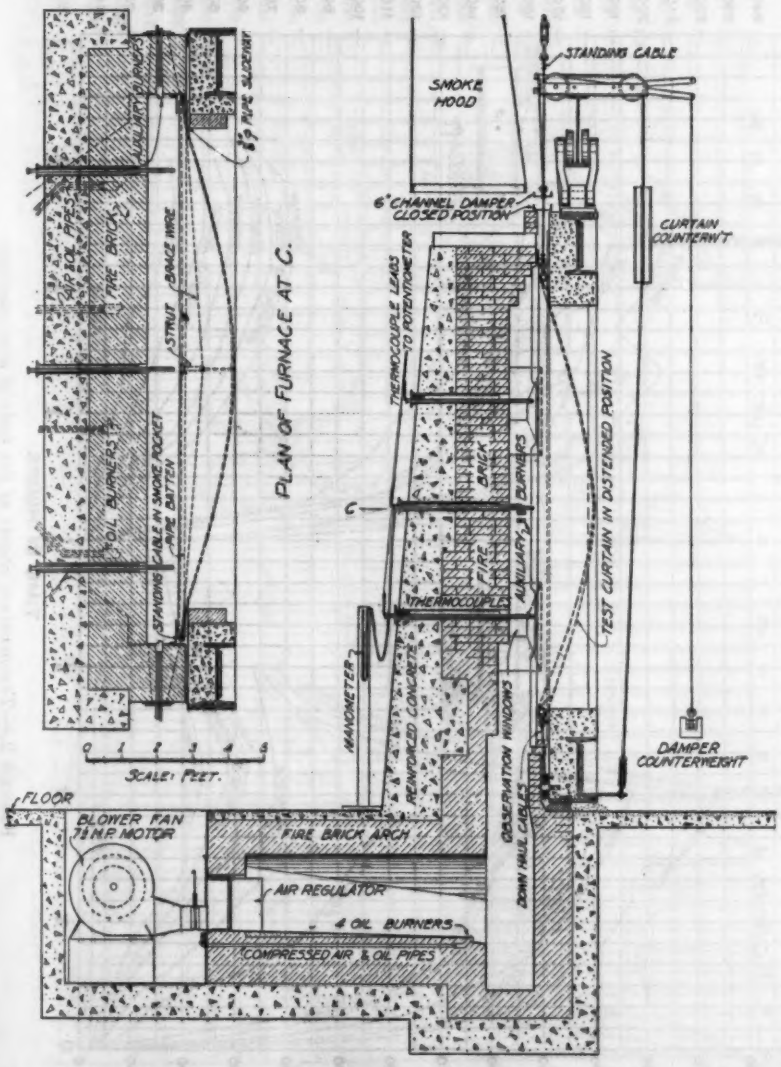


FIGURE 1.—*Vertical and plan sections of large panel furnace*

signed especially for these tests and were heavier and more substantial in every way than those usually installed in theatres. With one exception they had two thicknesses of cloth. Figures 4 to 9 also show some of the details of construction and the methods of mounting the curtains for the tests.

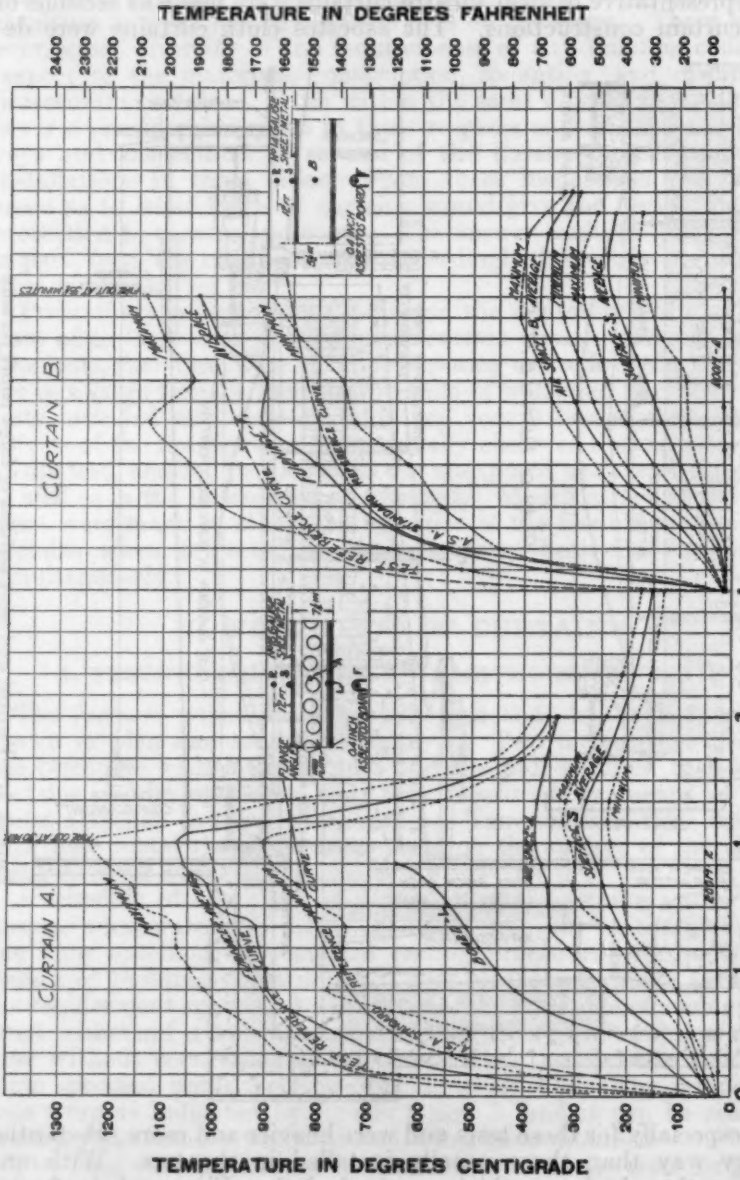


FIGURE 2.—*Temperature charts of fire tests of steel curtains*

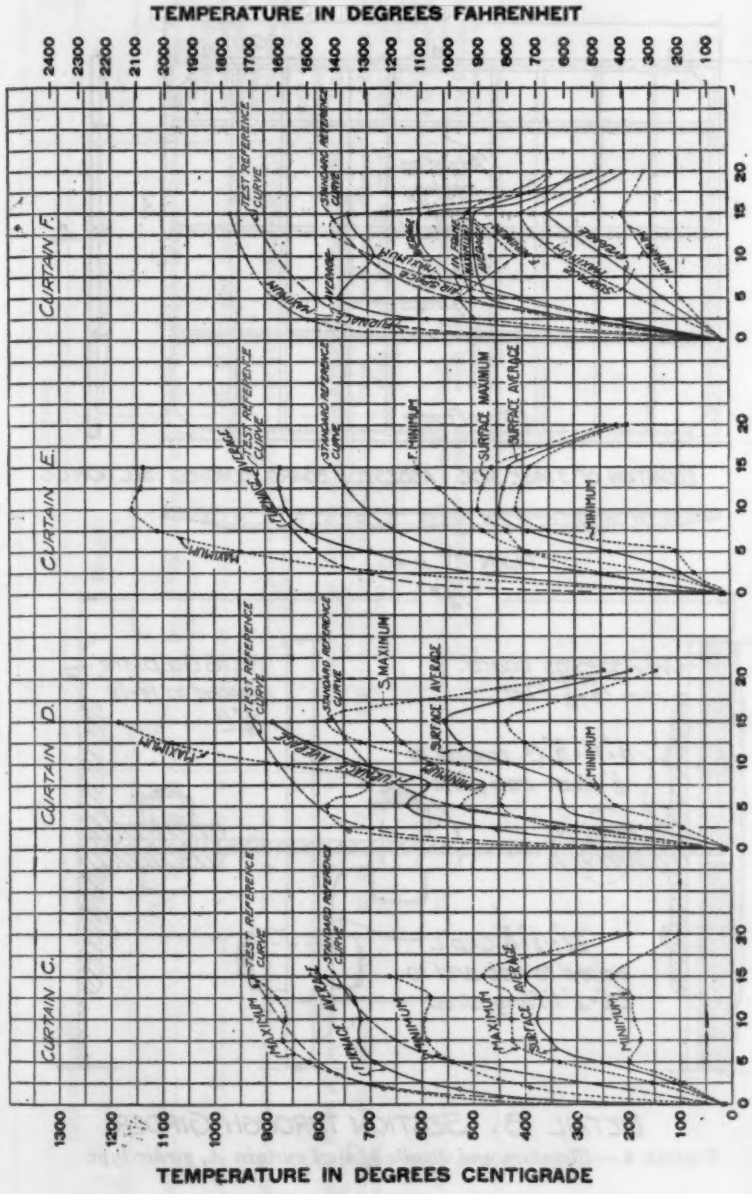


FIGURE 3.—*Temperature charts of fire tests of asbestos cloth curtains*

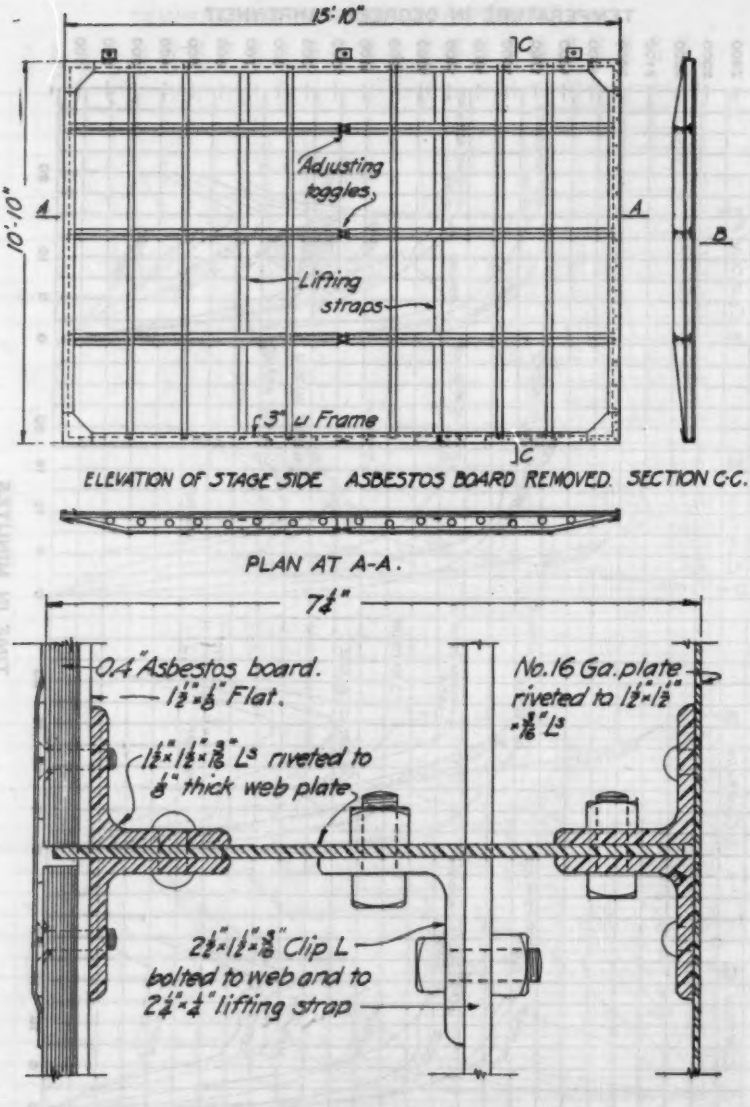


FIGURE 4.—*Elevation and details of steel curtain A, girder type*

TABLE 1.—*Details of test curtains*

Designation	Type of curtain	Size			Covering		General construction
		Width	Height	Thickness	Fire side	Unexposed side	
		Ft. in.	*Ft. in.*	*In.*			
A	Steel	15 10	10 10	7¼	0.4 inch thick asbestos millboard.	No. 16 gage steel plates	3-inch channel frame with 7-inch plate and angle girders placed horizontally.
B	do	9 0	6 0	5½	do	No. 14 gage steel plates	5 inches deep by 3 feet wide welded steel pans bolted together.
C	Flexible asbestos cloth curtain	16 6	12 0	¼	Single-ply plain asbestos cloth, 2.6 pounds per sq. yd.	Single-ply same as fireside	1¼-inch pipe battens top and bottom; three 5/16-inch diameter steel cables vertically between battens.
D	Flexible single ply asbestos cloth curtain.	16 6	12 0	1/16	Single ply of 4.6-pound asbestos cloth with Monel-Metal-wire reinforcement. Cables and battens covered with strips of cloth.		Do.
E	Semirigid 2-ply asbestos cloth curtain.	15 10	12 6	3¼	Asbestos cloth previously used in curtain *D*.	Single ply 3½-pound asbestos cloth with brass-wire reinforcement.	1¼-inch pipe across top, 5/16-inch cable in ½-inch pipe bottom batten. Sides, two 1¼ by 1½ by 5/16-inch angles and one 4 by ¼-inch plate.
F	do	15 4	12 0	3¼	Single ply 2¾-pound asbestos cloth with nickel-wire reinforcement.	Single ply 3¼-pound asbestos cloth with brass-wire reinforcement.	3-inch channel frame divided into 9 equal panels by 1¼-inch horizontal and vertical pipes.

Several factors other than heat transfer through the curtain by radiation and conduction influenced the results of the tests. Some of them were differences in pressure within the furnace chamber, the amount of combustibles in the fabric, or on the surfaces of the curtain, permeability to gases, and the closeness with which the edges of the curtain fitted the test frame.

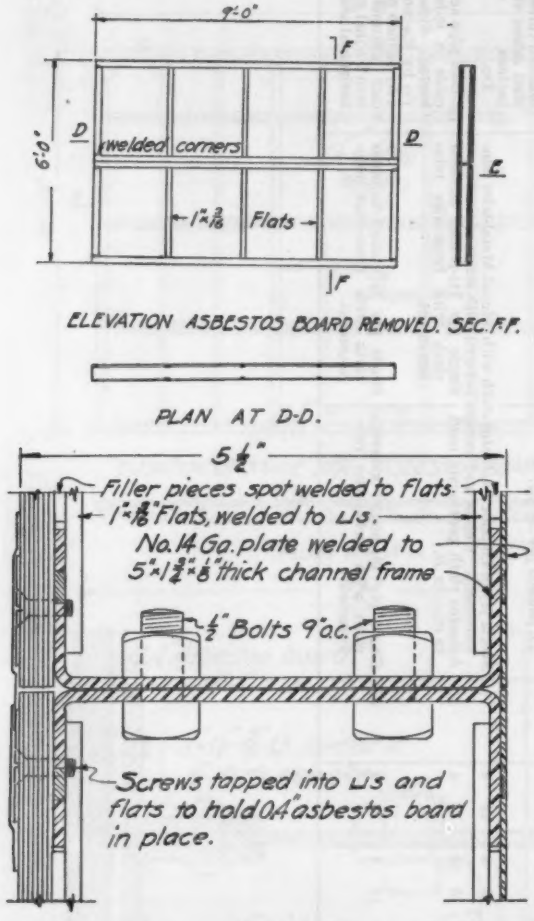


FIGURE 5.—*Details of steel curtain B, unit type.*

(a) CURTAIN A

This specimen curtain in all its essential details was like the full-sized curtains of this kind used in theatres. It consisted of a frame of 3-inch rolled channels and light plate and angle girders extending from side to side, with steel plates forming the face of the curtain and asbestos mill boards the back or stage side. The general structure of the test specimen is shown in figure 4.

There was excellent resistance to the passage of fire and smoke and no glow was visible on the front side of the curtain at any time during the test, notwithstanding some passage of flame around the edges and top of the curtain where the sheet metal fire stops warped out of place. The oil paint on the metal face of the curtain was charred black in some parts but during the test there was no appreciable amount of smoke.

During the test the curtain deflected a maximum of 1.94 inches towards the fire, equivalent to 7 inches center deflection in a 40-foot length, and there was local buckling of the faceplates. After the test the recovery of all parts to initial alignment was almost complete.

(b) CURTAIN B

This specimen consisted of two units such as are used to construct full-size curtains. The units were in the form of long pans, the edges of which were made of pressed steel channels welded to steel plates

which formed the auditorium face of the curtain. The flanges of the channels next to the other face were connected by flat bars at intervals of 3 feet to tie the opposite sides of the pans together and to support the edges of the 0.4-inch-thick asbestos boards. The assembly of the test specimen and its principal details are shown in figure 5.

The bottom and vertical edges of the curtain as mounted in the 8-inch brick wall built within the test frame were protected by

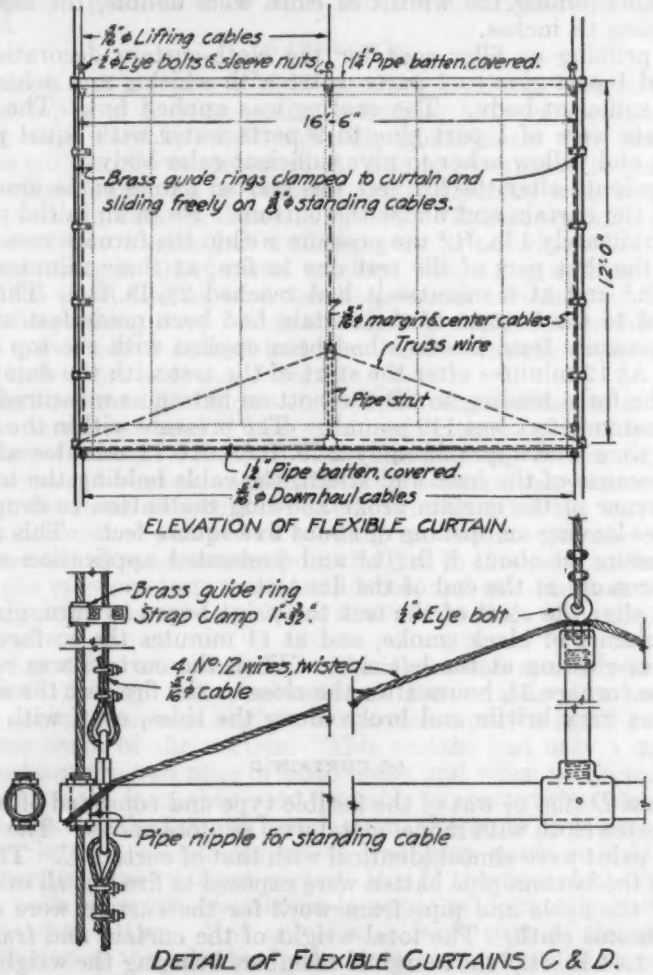


FIGURE 6.—*Details of asbestos cloth curtains C and D.*

mineral wool while the 1-inch space along the top was stopped by a sheet metal angle so that the top edge was exposed to the furnace fire.

Curtain *B* gave good performance in respect to holding back fire and heat and there was little local buckling of the face plates. The maximum center deflection in an 8-foot length was 0.3 inch, equivalent to 7½ inches in a 40-foot length. The recovery of the steel work from the deflections was as in the previous test, almost complete.

The asbestos boards on both curtains *A* and *B* buckled away from the fire at their centers, a few of them cracked, and all lost the greater part of their strength but remained in place during the test.

(c) CURTAIN C

This curtain was made of two separate curtains mounted on the pipe and cable frame shown in figure 6. The stitching twine was 3-ply asbestos reinforced with two no. 33 B. & S. gage brass wires. The seams joining the widths of cloth were double, the lap of the cloth being 1½ inches.

The priming or filler coat for the cloth-curtain decoration consisted of 1 part glue to 4 parts water with whiting and ocher added to give sufficient body. The coating was applied hot. The decorative coats were of 1 part glue to 9 parts water with equal parts of whiting and yellow ocher to give sufficient color body.

One minute after the fire test was started flames came around the sides of the curtain and under the bottom. From an initial pressure of approximately 1 lb./ft.^{2} the pressure within the furnace rose rapidly during the first part of the test due to fire; at three minutes it was 2¼ lb./ft.^{2} and at 6 minutes it had reached 2½ lb./ft.^{2} The cables attached to the bottom of the curtain had been made fast after the initial pressure from the fans had been applied with the top damper open. At 12 minutes after the start of the test with the damper still open, the force tending to lift the bottom batten as measured by the two dynamometers was 119 pounds. The pressure within the furnace at this time was approximately 2 lb./ft.^{2}. At 13 minutes after the start, because of the heat and strain, the cable holding the top right hand corner of the curtain broke allowing the batten to drop about 18 inches leaving an opening of about five square feet. This reduced the pressure to about ¾ lb./ft.^{2} and prevented application of additional pressure at the end of the fire test.

Soon after the start of the test the paint began to burn, giving off large volumes of black smoke, and at 11 minutes the surface of the cloth was glowing at the left side. When the curtain was removed from the furnace 3¾ hours after the close or the fire test the asbestos cloth was very brittle and broke along the sides, even with careful handling.

(d) CURTAIN D

Curtain *D* (fig. 6) was of the flexible type and consisted of a single-ply asbestos cloth with monel metal wire reinforcement. The mounting and paint were almost identical with that of curtain *C*. The wires trussing the bottom pipe batten were exposed to fire but all other portions of the cable and pipe framework for the curtain were covered with asbestos cloth. The total weight of the curtain and frame was 293 lb., 1.44 lb./ft.,^{2} the marginal members bringing the weight somewhat above the unit weights ordinarily encountered in practice.

Within a half minute after the start of the fire test, clouds of smoke came around the sides of the curtain and between the bottom batten and frame. The paint soon began to flake and fall off, the bare spots glowing briefly, probably because of the remaining combustibles. At 4½ minutes the paint had flaked off large areas of the curtain and the first steady glow of the curtain was observed at about five minutes. At 10½ minutes the curtain was glowing over about a third of its area and at 12 minutes more than half of the area was glowing brightly.

The glowing area increased until the end of the test. At 13 minutes 50 seconds the damper was closed and the pressure within the furnace increased to 4.1 lb./ft.^{2} without causing any breaks in the curtain. When pressure up to 3 lb./ft.^{2} was applied during the test, large quantities of gases and smoke leaked through the open mesh of the cloth.

Although this curtain had sufficient strength at the end of the fire test, the performance was in other respects not satisfactory. Dense smoke and glowing spots developed more quickly than with the doubleply curtain of plain asbestos cloth and the final glow was more pronounced.

(e) CURTAIN *E*

Curtain *E* had a frame of rigid members on the two vertical edges and the top and a comparatively flexible bottom member. The asbestos cloth curtain reinforced with monel-metal wire previously tested as curtain *E* was placed on the side to be exposed to fire. The unexposed side was of new asbestos cloth reinforced with brass wire. Details of the curtain are shown in figure 7. The side members, which consisted of two angles and a plate, were fitted with roller-bearing trolleys running in sheet metal tracks. The sizing and paint coats were of the same kind as used for the previous curtains, but had less glue in the priming coat. This curtain was designed and built after the two previously described had been tested with the object of overcoming their more serious defects.

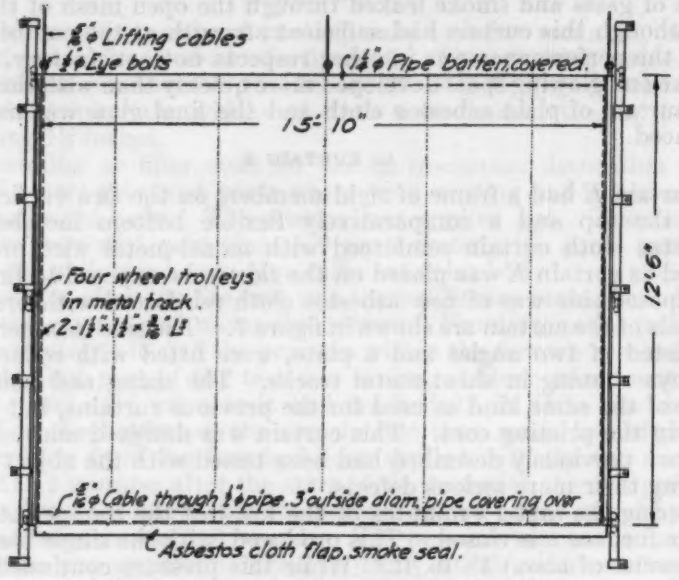
During the first 1½ minutes of the fire test the damper at the top of the furnace was closed. This produced, with the single blower fan, a pressure of about 1½ lb./ft.^{2} While this pressure continued, smoke and flame were forced through an opening between the curtain and the frame at the lower left corner. At 2 minutes after the start of the fire the decorative paint was burning on the face of the curtain. There was not as much smoke from the glue in this paint as from that used in the previous tests nor was it dense, and at 5 minutes after the start the smoke was very much less in evidence than while the damper was closed.

The second fan was started at 8½ minutes and sounds were heard as of cloth tearing. At the center of the curtain glow was soon noticed, which increased slightly but covered only a small part near the center seam of the curtain. This curtain had only a marginal frame to keep the two plies of cloth apart and when subjected to the pressure from the fans, they were probably in contact near the middle. The cloth next to the fire was found, after the test, to have a tear about 30 inches long at the crease which had been made in folding the cloth after its removal from curtain *D*. If the new cloth had been used on both sides for this curtain it would undoubtedly have withstood the test better. Not only had a large part of the water of crystallization of the asbestos in the cloth on the fire side been driven off in the previous test, but the cloth was also more open to the passage of gases.

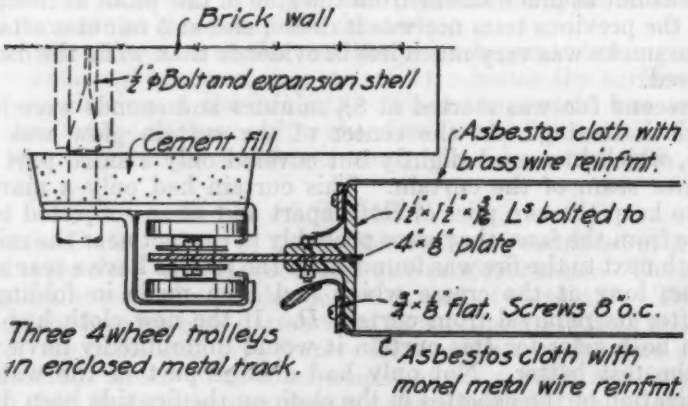
(f) CURTAIN *F*

The frame of curtain *F* was made of structural-steel shapes and pipes joined and trussed to form a relatively rigid structure on which were mounted the two plies of cloth (fig. 8). The seams were vertical and the plies were stretched tightly on the two sides and lapped around the edges of the frame to form an extra protection for the

channel. While in actual installations asbestos cloth curtains having pipe frames of semirigid construction are mounted on I-beam tracks with ball-bearing trolleys attached to the sides of the frame, in the test the trolleys were replaced by guides cut from ⅜-inch steel plates. A


⅝" ϕ Lifting cables
¾" ϕ Eye bolts
1¼" ϕ Pipe batten covered.
15'-10"
12'-6"
Four wheel trolleys in metal track.
2-1½" × 1½" × ⅜" L's
⅝" ϕ Cable through 1" ϕ pipe. 3" outside diam. pipe covering over
Asbestos cloth flap, smoke seal.

ELEVATION OF SEMI RIGID CURTAIN E


Brick wall
½" ϕ Bolt and expansion shell
Cement fill
Asbestos cloth with brass wire reinf'mt.
1½" × 1½" × ⅜" L's bolted to 4" × ⅜" plate
¾" × ⅛" flat. Screws 8" o.c.
Three 4 wheel trolleys in enclosed metal track.
Asbestos cloth with monel metal wire reinf'mt.

DETAIL OF SEMI RIGID CURTAIN E.

FIGURE 7.—*Details of curtain E.*

rigid type of frame mounted with sliding shoes on guide rails, resulting in less pull against the guides and proscenium wall than with the semirigid frame with the trolley mounting, is more generally used in recent installations.

FIGURE 9.—*Curtain F during fire test. Smoke leaking under bottom.*

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The sizing and decorative coats were a casein and sodium silicate paint developed for the purpose because of the unsatisfactory behavior of the paints made with animal glue.

The fire test was conducted in the same manner as the others except that the air pressure was less. A small amount of smoke issued from under the lower left corner of the curtain at 1 minute 47 seconds after the start of the test because of a deficient bottom seal. At 1 minute 50 seconds the paint on the outer face of the curtain began to smoke and turn a reddish brown color and at 5¼ minutes (fig. 9) nearly all the surface was brown. The smoking of the face of the curtain was not serious but there was leakage of flame and smoke along more than half the length at the bottom. The rise of temperature of the furnace had been more rapid than for the previous tests of asbestos cloth curtains. At 8 minutes the smoking

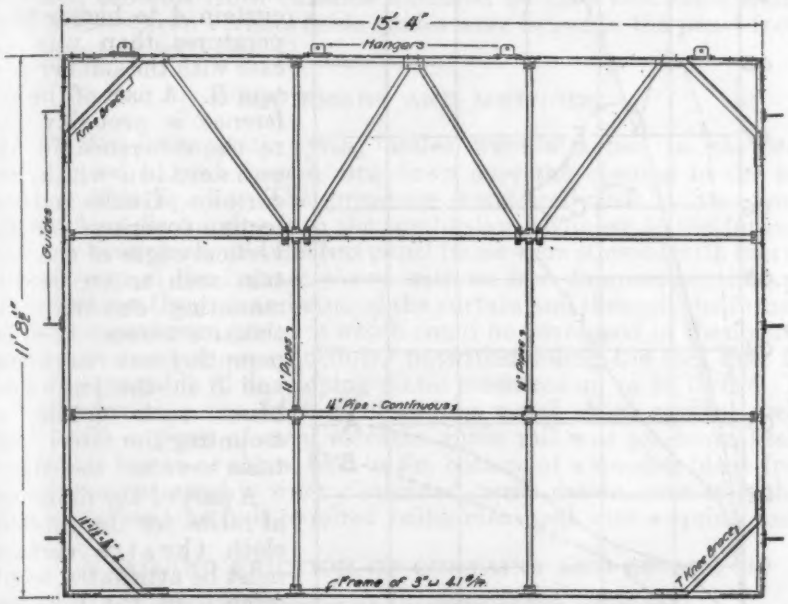

FIGURE 8.—*Details of the frame of curtain F.*

of the surface had ceased and within another minute there was no smoke coming under the curtain. At 9 minutes the flame was passing downward between the top of the curtain and the test frame, causing glow at this opening. At 12 minutes there was a slight glow in the lower right corner of the panel where the paint had not entirely filled the interstices in the cloth. At the end of the 15-minute test no more glow was visible than at 12 minutes. The expansion of the steel members sheared off the ½-inch diameter bolts and rivets fastening the ends of the guide rails and guard plates at the edges of the curtain. There was no passage of flame around the vertical edges of the curtain, but a better closure along the bottom and top than this curtain had during the test was indicated as desirable. The results with this curtain were better than those obtained with the three other asbestos curtains mainly because of the positive separation between the plies

of the cloth, the better sealing of the vertical edges against the passage of flame, and the use of a decorative paint having only a small proportion of organic matter.

(d) COMPARATIVE PERFORMANCE

The resistance of the several curtains to the transmission of heat can be judged qualitatively by comparing the ratios of the rise of the average surface temperature during any period from the beginning to the average furnace temperature above its initial temperature maintained during thet period. The values of this ratio, R, for all six of the curtains have been plotted in figure 10. The difference between the curves for curtains A and B can be attributed largely to the exposure of the edges of curtain A to higher temperatures than was the case with the smaller curtain B. A part of the difference is probably due to the difference in the convection within the curtains. Guides and protective coverings for the vertical edges of the curtain such as are used in mounting curtains in theatres werenot used for mounting test curtains A and B in the furnace; hence such details of mounting for steel curtains were not tested.

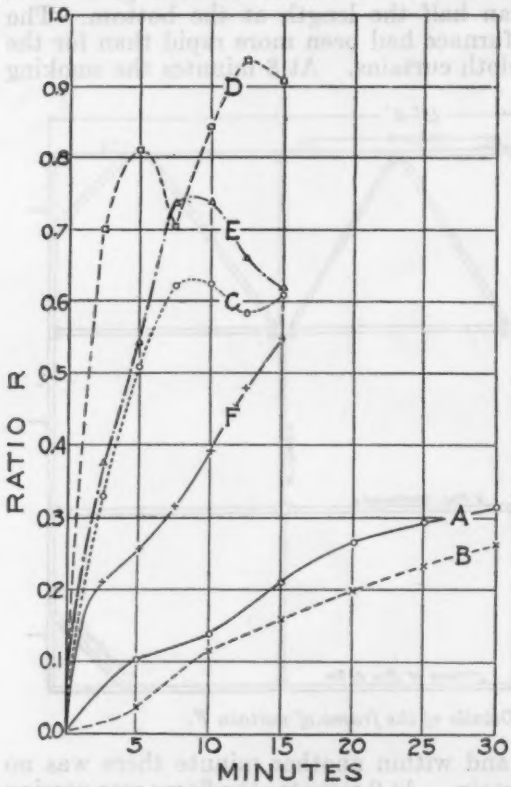

FIGURE 10.—*Ratio of rise of surface temperature of curtain to averge maintained temperature of furnace above its initial temperature.*

A part of the difference of ratios for the asbestos cloth theatre curtains must be attributed to difference in the temperatures in different parts of the furnace relative to the points where measurements on the surface of the curtain were made. In general, however, the ratio gives a fair measure of effectiveness in resisting heat flow.

With respect to the protection afforded by the steel curtains, the tests herein reported confirm the results of tests made at Underwriters' Laboratories, Chicago, soon after the Iroquois Theatre fire. Regarding these tests, John R. Freeman in his paper, "On the Safeguarding of Life in Theatres", says: "It was plain to all who witnessed these tests that the sheet-steel curtain, protected with some asbetic material on the fire side, possessed far greater strength than the simple asbestos. The thin sheet of steel, moreover, cut off the view

of the fire that was apparent through the texture of the asbestos canvas.

"With care given to the design of the guides and fastenings at edges and top, so that after it was lowered the curtain could not be pulled out by warping, buckling, smoke explosions, or pressure of air, the steel curtains would have a value to the fire underwriter that no asbestos curtain can possess, and would probably hold a fire on the stage from entering the auditorium."[2]

III. OPERATING TESTS OF CURTAINS

Since some of the theatre curtains inspected during the survey would not close when subjected to small lateral forces, it was thought advisable to investigate this phase of the problem. Accordingly, the two asbestos cloth curtains mounted on pipe and cable frames and held between vertical cable guides were rigged in the panel frame for operating tests (fig. 1).

1. EQUIPMENT AND METHODS

Two 500-lb.-capacity spring scales were attached to the steel cables used to pull the curtains down over the opening in the test panel. (See fig. 1.) The pressures were produced by two small blower fans discharging into the combustion chamber of the furnace. All the openings around the test panel frame were stopped with mortar and the top of the furnace closed with an iron damper; but because of air leakage through and around the curtain and through the furnace walls the maximum pressure which could be developed in the furnace against the curtain was 4.6 lb/ft.^{2} notwithstanding the fact that the fans were capable of developing static pressures up to 25 lb/ft ^{2}.

For measuring the friction of sliding metal shoes against metal guide rails a piece of steel elevator guide rail was set horizontally and metal blocks or shoes, held in the bottom of a wooden block from which various weights were suspended, were drawn over it with a small windlass, the pull required being measured with a spring scale.

2. SLIDING FRICTION OF CURTAINS AND GUIDES

(*a*) OPERATING TESTS OF ASBESTOS CLOTH CURTAINS

The operating tests were planned to determine the friction when air pressure was applied to the asbestos cloth curtains. The guide rings on the vertical edges of the test curtains ran smoothly on the guide cables and the counterweights were adjusted so that little force was required to lower or raise the curtains. In the first tests with curtain *C* the cloth was allowed to rub against the brick and concrete surrounding the opening over which the curtain was hung. Afterwards slideways made of three-eighths inch iron pipe were provided at the vertical sides of the opening to reduce the friction. It will be observed that such slideways were not found in actual installations but were introduced here to determine the decrease in friction.

Coefficients of friction varied widely, but the average ranged between unity and one half. With pipe slideways the coefficients of friction were less than two thirds those found in the first tests.

[2] On the Safeguarding of Life in Theatres, John R. Freeman, Trans. A.S.M.E., vol. 27, 1906.

The large friction factors found in these tests were in keeping with the observations made in connection with the operation of curtains in theaters, where slight lateral pressures arrested the descent of curtains of this type.

The raising and lowering of curtain *E* showed that the roller-bearing trolleys moved within the closed track very easily, but due to the lack of internal diagonal bracing the curtain would bind when one end was lowered faster than the other. The time allowed for the tests did not permit correction of this defect and therefore the operating tests and friction measurements could not be made. As trolleys were not used in mounting curtain *F*, no tests were made to measure the sliding friction of this curtain.

(*b*) RESULTS OF FRICTION TESTS OF METAL BLOCKS ON GUIDE RAILS

The tests of metal blocks sliding on steel rails gave widely varying results, as it was not possible to secure guide rails with surfaces of uniform frictional properties throughout their length. The values in table 2 have been selected as suitable for use in the design of curtains and curtain rigging. The factors for rolling friction have been taken from other sources,[3] while those for sliding friction were taken from the results of the tests and in most cases have been checked against values reported by others.

TABLE 2.—*Friction of proscenium curtain guides*

Surface	Lubrication	Mill rolled steel rail [1]		Machined steel rail	
		Coefficient of friction	Required underbalance, pound per square foot of opening	Coefficient of friction	Required underbalance, pound per square foot of opening
Roller or ball bearing wheel trolleys	Oiled	0.03	[2] 0.15	0.025	[2] 0.125
Metal sliding shoes on steel guide rails	None	.25	1.25	.20	1.00
	Greased	.12	0.60	.10	.50
Asbestos cloth on rounded steel rail	None	.65	3.25		
Asbestos cloth on rough brickwork	do	0.8 to 1.0	4.0 to 5.0		

[1] The underbalances are the difference in weight of the curtain, including chains and lifting cables below sheaves, and the counterweight with its fastenings, cables, etc. The underbalance should be proportioned to the size of the curtain opening and should vary with the type of curtain mounting used. A lateral pressure on the curtain during closure of 5 pounds per square foot is assumed.
[2] It is recommended that underbalance less than 0.2 lb/ft. [3] never be used.

IV. FIRE TESTS WITH DECORATIVE PAINTS

The three curtains, *C*, *D*, and *E*, were unsatisfactory in that large volumes of dense smoke were given off from burning paint and combustibles in the cloth. Smoking began soon after the fire was lit in the first two tests and was in such quantity in all three tests that it might have caused a panic if coming from a curtain in a theater. A paint which would give off less smoke was therefore sought.

[3] Mechanical Engineer's Pocket Book, Kent, 10th Edition.

1. TESTING METHODS AND RESULTS

(a) BUNSEN-BURNER TESTS

Eighteen tests were made by painting pieces of reinforced asbestos cloth with dyes, casein, animal glue, or sodium silicate paints, and with combinations of some of them, and subjecting limited areas to a bunsen flame. The dyes gave the most pleasing appearance and the least smoke, but the combination of casein and sodium silicate with mineral pigments, although giving more smoke than the dyes, seemed more satisfactory from the standpoint of filling the interstices of the cloth.

(b) PANEL TESTS

Six 5- by 40-inch panels were then prepared so that comparison of the smoke given off could be made by exposing three parallel panels to a furnace fire at a time. The three panels were painted on a single piece of cloth and exposed directly to the furnace fire in one test. In the other test two plies of cloth were used, one on which the panels were painted and the other, filled by painting with two coats of sodium silicate and mineral pigment, was interposed between the test panels and the fire.

As in the small tests, the dyed panels again gave the least smoke, but the glow of the fire could be seen through the open mesh. The paint in which animal glue was used gave a black smoke and in the greatest volume. The paint composed of casein, sodium silicate, whiting, and ocher gave a bluish-colored smoke intermediate in volume between that from the other two panels. This paint was adjudged the most suitable of the several tried. The most satisfactory proportions tried, those used on curtain *F*, were as follows:

4 pounds powdered casein.
½ pound carbonate of soda.
10 pounds sodium silicate solution (commercial water glass, specific gravity 1.38).
20 pounds whiting and powdered yellow ocher mixed.
Water sufficient to give the desired consistency.

V. TESTS OF ASBESTOS CLOTH

1. STRENGTH AND STRETCH TESTS

(a) EQUIPMENT AND METHODS

Gas flames were used to heat the cloth in trial tests, but, as neither control nor measurement of temperature was satisfactory, electric heating was adopted. The necessary heating elements and temperature-measuring devices were added to a Scott horizontal textiles testing machine. Temperatures were measured by means of chromel-alumel thermocouples and a portable potentiometer. Stretch of cloth was measured by means of a scale and a pointer attached to the specimen at points 3 inches apart, after an initial pull of 3 pounds had been applied.

The specimens for strength and stretch tests were all cut 6 inches long by 1⅛ to 1¼ inches wide and then raveled to a definite number of threads in width for 1 inch as determined by thread counts over 5 inches or more width of cloth. For those specimens which were stronger at the elevated temperatures it was necessary to notch each side at the center to obtain the break within the heated region.

(b) ASBESTOS CLOTHS

Eleven of the twelve samples of asbestos cloths tested were of white, chrysotile asbestos, a hydrous silicate of magnesia, and one was of the African or Cape blue, crocidolite asbestos, an iron-sodium silicate.[4]

Eight samples had wire reinforcement in the yarns. The characteristics of the various cloth samples are given in table 3.

TABLE 3.—*Characteristics and strength of asbestos cloths*

Sample no.	Weight per square yard	Kind of cloth and reinforcement per strand	Number of strands per inch		Area of wire per strand	Strength per inch (width)		Strength per strand		Strength,[1] weight ratio	
			Warp	Filling		Warp	Filling	Warp	Filling	Warp	Filling
	Lb.				*Sq. in.*	*Lb.*	*Lb.*	*Lb.*	*Lb.*	*Lb.*	*Lb.*
1	2.44	Plain	21	11		88	40	4.2	3.6	35.7	16.0
2	3.05	Reinforced two no. 36 brass	14½	10½	0.00002	161	77	11.1	7.3	52.8	25.2
3	2.29	Plain African blue	18	7		159	31	8.8	4.4	69.5	13.5
4	4.31	Reinforced two no. 30 monel	16	9	.000068	302	175	18.9	19.4	70.0	40.7
5	3.06	Reinforced one no. 30 nichrome	16	9	.000072	212	137	13.2	15.2	69.3	44.8
6	5.21	Reinforced one no. 30 nichrome	16	8	.000072	353	199	22.1	24.9	67.9	38.2
7	2.53	Plain	20	11		141	82	7.0	7.5	55.7	32.4
8	4.61	Reinforced two no. 32 monel curtains *D* and *E*	16	7	.000048	312	116	19.5	16.6	67.7	25.1
9	3.28	Reinforced two no. 32 brass wires	17	9	.00005	206	113	12.1	12.6	63.4	34.4
10	2.77	Reinforced two no. 34 nickel in warp. One no. 34 in filling	17	8	.00003	213	95	12.5	11.9	76.9	34.2
11	3.14	Reinforced two no. 33 brass	16	8	.000044	198	92	12.4	11.5	63.1	29.3
12	3.87	Plain	21	11		84	30	4.0	2.7	21.7	7.8

[1] The ratio is derived by dividing the strength (lb./in. width) by the weight (lb./yds.²).

(c) STRENGTH TESTS AT ORDINARY TEMPERATURES

The tests were made at room temperature which varied from 70° to 80° F. (21° to 29° C.). The values found show large variations, but for direct comparison of strength the last two columns of table 3 are most significant.[5] In these columns the relation of strength to weight of cloth has been given. While some of the white (chrysotile) plain asbestos cloths show low strength compared to the wire-reinforced cloths, on this basis the warps of the blue (crocidolite) plain asbestos cloth compared favorably in strength with the reinforced chrysotile cloths.

(d) STRENGTH AT ELEVATED TEMPERATURES

The strength of most of the samples listed in table 3 were also determined when heated in an electric furnace. The results are given in pounds per inch width in figure 11. There was an increase of strength of chrysotile asbestos cloths with increase of temperature up to approximately 300° C. (572° F.) after which there was a rapid

[4] The reader is referred to Non-Metallic Minerals, by Raymond B. Ladoo, for information on the occurrence, mining, preparation, and uses of the several asbestos minerals.

[5] Compare with values given by Freeman, "On the safeguarding of life in theaters." Trans. A.S.M.E. vol. 27, p. 110, 1906.

reduction of strength with further increase of temperature. The white unreinforced asbestos cloths when heated above 400° or 500° C. (752° or 932° F.) lost a considerable part of their strength, but the wire-reinforced cloths lost comparatively little strength until temper-

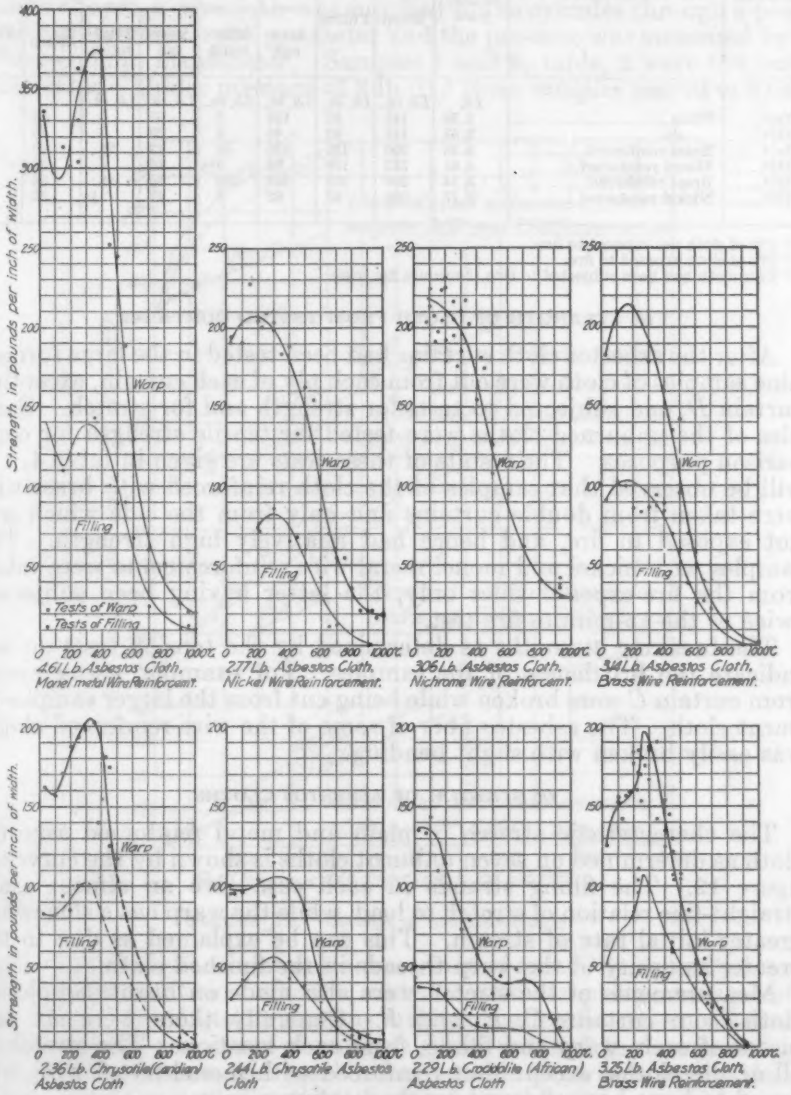
Strength in pounds per inch of width.
Warp
Filling
Tests of Warp
Tests of Filling
4.61 Lb. Asbestos Cloth, Monel metal Wire Reinforcement.
2.77 Lb. Asbestos Cloth, Nickel Wire Reinforcement.
3.06 Lb. Asbestos Cloth, Nichrome Wire Reinforcem't.
3.14 Lb. Asbestos Cloth, Brass Wire Reinforcement.
2.36 Lb. Chrysotile (Canadian) Asbestos Cloth
2.44 Lb. Chrysotile Asbestos Cloth
2.29 Lb. Crocidolite (African) Asbestos Cloth
3.25 Lb. Asbestos Cloth, Brass Wire Reinforcement.

FIGURE 11.—*Strengths of asbestos cloths when subjected to heat in an electric furnace.*

atures exceeding 480° C. (906° F.) had been reached. There was marked contrast between some of the plain and reinforced cloths in this respect. Blue asbestos cloth lost strength after temperatures of 100° C. (212° F.) had been exceeded. The strengths of both plain and reinforced cloths were small at temperatures above 900° C. (1,652° F.).

TABLE 4.—*Strength of cloth samples cut from tested curtains*

Curtain	Kind of asbestos cloth	Weight per square yard	Strength before test		Strength after test				Percent of original strength remaining	
			Warp	Filling	Warp		Filling			
					Average	Minimum	Average	Minimum	Warp	Filling
		Lb.	*Lb. in.*	*Lb. in.*	*Lb. in.*	*Lb. in.*	*Lb. in.*	*Lb. in.*		
C(*a*)[1]	Plain	2.53	141	82	125	3	62	3	89	75
C(*b*)[2]	do	2.53	141	82	46	3	29	3	33	35
E(*a*)[1]	Brass reinforced	3.28	206	113	135	64	72	26	66	66
E(*b*)[2]	Monel reinforced	4.61	312	116	83	9½	46	4	27	40
F(*a*)[1]	Brass reinforced	3.14	206	103	191	130	85	48	93	82
F(*b*)[3]	Nickel reinforced	2.77	236	87	82	5	42	1½	35	48

[1] Ply of cloth not exposed to fire.
[2] Ply of cloth exposed to fire.
[3] This cloth had been subjected to two 15-minute fire tests.

(e) STRENGTH OF CLOTH FROM TESTED CURTAINS

After the asbestos cloth curtains had been tested in the large furnace nine samples of cloth were cut from each ply of each curtain, excepting curtain *D*, and subjected to tests for strength and for stretch. Samples of the unburned cloths were tested for tensile strength for comparison purposes. The results of these tests are given in table 4. It will be observed that samples of the cloth reinforced with brass wire were taken from double curtains and only from the side which was not exposed to fire, and hence had relatively high strength. The samples with nickel and monel metal wire reinforcements were taken from the fire-exposed sides only, the latter having been subjected twice to the 15-minute fire test.

The breaking strengths as determined by the tension tests do not indicate the brittleness of the samples. For example, many pieces from curtain *C* were broken while being cut from the larger samples of burnt cloth. The asbestos fiber of some of the wire-reinforced cloths was easily broken with slight bending.

(f) STRETCH OF ASBESTOS CLOTHS

The characteristic stretch of plain and metal reinforced asbestos cloth as determined on seven unburnt cloths is shown by the curves in figure 12. The filling strands in each case give an almost exact straight-line relation of stretch to load, while the warp has a somewhat greater initial rate of stretch. This can be explained as due to the greater sinuosity of the warp threads in the finished cloth.

Measurements of the stretch were also made on burnt samples of cloth from curtains *C*, *E*, and *F*. Generally there were six test pieces of each, warp and filling, from each location. The stretch of all of the cloths, except that reinforced with monel-metal wire, was found to have been reduced by the heating.

2. PERMEABILITY TO AIR

Tests for permeability to air were made on samples of cloth with pressures from 1/2 to 5 lb./ft.^{2} Some of these cloths were also given two coats of paint to fill the interstices to determine what improvement in resistance to air flow was obtained.

The apparatus for measuring permeability to air consisted of a cylinder with a flanged end to which a sample of the cloth could be clamped with the other half of its companion flange. The openings through the flanges were of two sizes. One was of 22.7 in.^{2} area for testing the less permeable specimens, and one of 5.7 in.^{2} for testing the cloths of open weave. Air was supplied to the cylinder through a positive displacement type gas meter and the pressure was measured by a water-column manometer. Samples 1 and 6, table, 3 were the least permeable. Under pressure of 2 lb./ft.^{2} these samples passed one half

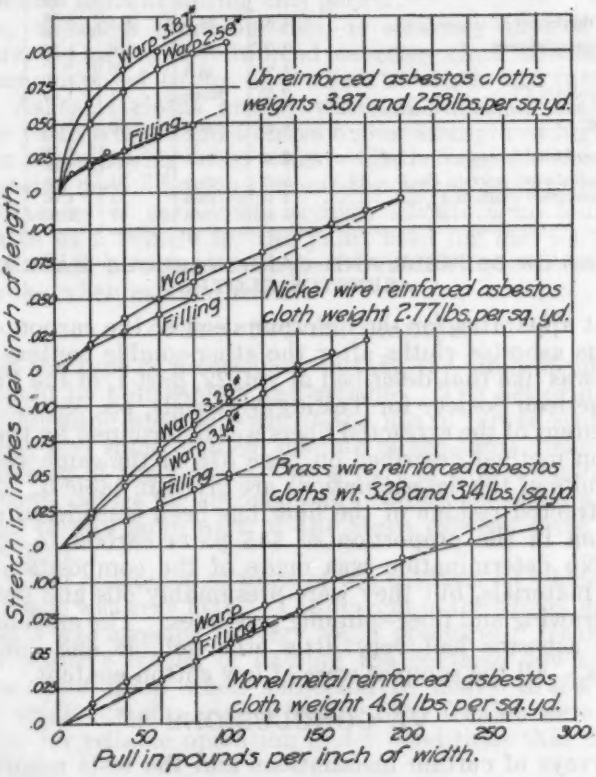

FIGURE 12.—*Stretch of asbestos cloths.*

cubic foot of air through each square foot of area. Samples 7, 8, and 12 passed about twice as much, and the other samples passed quantities ranging from 7 to 35 times as much air under the same conditions. Painting with two coats of suitable fillers reduced the permeability, at 2 lb./ft.^{2} pressure, to 1½ cubic feet per minute, which is considered satisfactory for service in theater curtain installations.

The paint used on curtain *F* was found to be effective for the purpose. Many paints other than those used would, undoubtedly, be suitable for filling the cloth. However, casein or other pliant binder to prevent cracking and dusting is essential with the more open cloths.

TABLE 5.—*Analyses of asbestos cloths*

Sample no.	Kind of cloth	Weight	Reinforcing metal	Extracted by ether	Carbon in residue of fibers	Cotton in fibers computed	Cotton in cloth computed
		lb./sq. yd.	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
1	Plain chrysotile	2.44	None	1.9	1.60 1.68	3.6 3.8	3.7
2	Brass reinforced	3.05	25.0	1.2	1.875 1.875	4.2 4.2	3.2
3	Plain crocidolite (African blue)	2.29	None	.2	.135 .135	.3 .3	.3
4	Monel reinforced	4.31	39.8	1.5	1.64 1.68	3.7 3.8	2.25
5	Nichrome reinforced	3.06	27.1	1.0	1.84	4.1	3.0
6	do	5.21	16.3	.5	1.70 1.70	3.8 3.8	3.2
7	Plain (curtain *C*)	2.53	None	.7	1.09	2.5	2.5
8	Monel metal reinforced (curtains *D* and *E*)	4.61	23.4	.6	1.15 1.03	2.6 2.3	1.87
9	Brass reinforced (curtain *E*)	3.25	34.3	1.1	1.12 1.04	2.50 2.35	1.59
10	Nickel reinforced (curtain *F*)	2.77	28.9	.4	.83	1.86	1.32
11	Brass reinforced (curtain *F*)	3.14	35.6	.3	1.06	2.4	1.54
12	Plain	3.87	None	.7	1.09	2.5	2.5

3. TESTS FOR SOLUBLE AND CARBONACEOUS MATERIALS IN ASBESTOS CLOTHS

The test apparatus for the determination of the carbon content of the various asbestos cloths after the ether-soluble content had been extracted was like that described in vol. 22, part I, of the Proceedings of the American Society for Testing Materials, pp. 580–582, and the carbon content of the extracted fibers was determined by the standard combustion method described on page 578 of the same volume.

The results of the determinations are given in table 5. All carbon on the extracted residue of the fiber has been considered as derived from cotton in the proportion of 445 parts carbon to 1,000 parts cotton. No determination was made of the composition of ether-extracted materials, but they were presumably oils and greases from the wire-drawing and fiber-spinning processes. The crocidolite (African blue) asbestos had very little ether-soluble and carbonaceous substances. All the samples were of low cotton content.

VI. CONCLUSIONS

The surveys of curtain installations and the tests reported herein have led to the following observations with respect to the several types of curtains and their effectiveness. Recommended requirements for curtain installations have been formulated.[6]

1. FIRE RESISTANCE

The purpose of the fire-resistive curtain is to close the proscenium opening against the passage of fire and smoke into the auditorium in dangerous or alarming amounts. The single-ply asbestos cloth curtain as found in many installations might screen stage fires from the audience under the less severe conditions, but unless supplemented by effective vents above the stage would permit ready flow of smoke and gases into the auditorium. They would show glow quickly if subjected to flames. Drafts and light winds lift the botton from the floor and moderate air pressures or winds might tear them from their

[6] Copies of "Recommended Requirements for Theater Proscenium Curtains" may be obtained by application to the Bureau of Standards, Washington, D.C.

mountings. None were adjudged to give adequate closure when exposed to severe fire or draft conditions.

Proscenium openings can be adequately closed against severe fires for periods of 15 minutes with a curtain composed of two plies of 3¼ pound wire-reinforced asbestos cloth with positive separation between the plies, such as in test curtain *F*, and of sufficient strengh to resist the pressures encountered under fire conditions.

Steel curtains of the types tested form effective barriers against fire for one half hour or more and do not glow or pass smoke and gases in objectionable amount during this period.

Asbestos, although incombustible, is seriously injured by high temperatures. Only wire-reinforced asbestos cloth should be used for structures exposed to fire and where strength and integrity are essential. Asbestos cloths having wires of nickel or nickel alloys spun in the yarns were found to have higher strength at high temperatures than those having brass wires. Cloths used for theater proscenium curtains need filling to prevent the flow of smoke through the weave. Mixtures of casein and sodium silicate were found to be more suitable as a vehicle for the paint used for this purpose than animal glue which has been much used but which gives off quantities of dark smoke when exposed to fire.

2. OPERATING MECHANISM

Whatever the type of proscenium curtain, the mechanism for operating it should be both simple and reliable. The better devices for emergency lowering operate without the use of power sources other than gravity, and some of them operate in the same manner for the regular lowering of the curtain. As a part of this mechanism there should be some device for automatically closing the curtain when the temperature of any large part of the stage exceeds some predetermined safe limit.

The frictional resistance of some of the curtains on which observations were made was very high. The descent of the single-ply asbestos cloth curtains was arrested by very slight pressures. Heavy bottom battens or positive means of hauling the curtains down would be required to close them when subjected to heavy drafts or wind. Except for small curtains, guides having little friction were indicated as necessary for reliable operation under conditions that may occur with fire.

Whatever the type of curtain or operating mechanism, reliable means of ventilation for the discharge of flame, smoke, and gases are necessary to the safety of the audience in the event of serious stage fires.

VII. ACKNOWLEDGMENTS

Acknowledgment for assistance is given to the Elevator Supplies Co., Hoboken, N.J., and James H. Channon Manufacturing Company, Chicago, Ill., for supplying the steel curtains; to Keasbey and Mattison Co., Ambler, Pa., and Peter Clark, Inc., New York City, who supplied asbestos cloths and the steel frame and mounting for the rigid double asbestos cloth curtain. The author also makes acknowledgments to the members of the fire-resistance section of the Bureau of Standards for their cooperation and to Messrs. W.H. Smith and T.P. Sager of the chemistry division for analyses of samples of asbestos cloths.

Washington, D.C., July 8, 1933.

U.S. Department of Commerce, Bureau of Standards

RESEARCH PAPER RP604

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

THE DEPOSITION OF CHROMIUM FROM SOLUTIONS OF CHROMIC AND CHROMOUS SALTS

By Charles Kasper

ABSTRACT

The confusing and apparently contradictory literature on the electrodeposition of chromium from trivalent and bivalent salts was critically examined and pertinent experiments were performed.

The main obstacle to obtaining bright deposits at high efficiency is not, as formerly supposed, in keeping the solutions of trivalent chromium violet; that is, preventing the formation of green undissociated molecular ions; but in maintaining a low concentration of hydrogen ions and a high concentration of chromium ions in the cathode film. The use of chromous salts or of chromic complexes does not circumvent this difficulty. The chromic acid bath is inherently superior to those containing chromium in the lower valence states.

CONTENTS

		Page
I.	Introduction	515
II.	Historical review	516
III.	Baths containing simple trivalent salts	518
	1. Theory	518
	2. Experimental work	520
IV.	Baths containing chromous salts	522
V.	Baths containing complex chromium compounds	524
VI.	Comparison of results with chromic acid bath	524
VII.	Conclusions	525
VIII.	Acknowledgments	525
IX.	Bibliography	525

I. INTRODUCTION

All commercial chromium plating is conducted in chromic acid baths, in which the chromium is sexivalent and is deposited with a low cathode efficiency. Bright deposits are obtained only over a limited range of current density and the throwing power is poor. It has been believed that this relatively poor performance is associated with the fact that the deposition occurs from the higher state of oxidation, and that better results would be obtained with lower states of oxidation. A bath of the latter type would more closely approximate the usual plating bath, whose performance leaves little to be desired in the case of nickel, zinc, and iron. A large number of investigations have been made on the deposition of chromium from the lower states of oxidation, but no such baths are at present in commercial use.

The purpose of this investigation was to consider critically the contradictory literature on this subject and to make further experiments that appeared to have promise. To warrant adoption, any

new type of chromium bath should be at least equal, or preferably superior, to the chromic acid bath in the following respects: (1) It should produce deposits that are bright, or which can readily be buffed bright; (2) it should produce such deposits over a fairly wide range of temperature and current density, and preferably at room temperature and at a relatively low current density; (3) the cathode efficiency should be at least equal to 15 percent as calculated for sexivalent chromium (which is equivalent to only 7.5 percent for trivalent or 5 percent for bivalent chromium); (4) the baths should be inexpensive, simple, stable, and easily controlled.

Failure to describe accurately the deposits obtained probably accounts for some of the contradictions in the literature, and especially some of the very high cathode efficiencies that have been reported for good plate. Dark, spongy, or impure deposits are practically useless for electroplating. Although the above requirements are very moderate, especially in view of the limitations of the present chromic acid baths, the net result of this survey and research is the conclusion that they are not met by any bath thus far developed, and there is little reason to believe that they can be met.

II. HISTORICAL REVIEW

Chromium was first deposited from the trivalent state by Junot (1).[1] Bunsen (2) worked with a chromous-chromic bath in a porous pot and obtained a bright deposit at an elevated temperature and a high current density. He emphasized the importance of the chromous ion. Placet and Bonnet (5) patented the use of trivalent chromium baths with lead anodes. In some of their patents the addition of alkali or alkaline earth chlorides was specified. Moeller and Street (3) worked at elevated temperatures with a concentrated solution of chrome alum to which a large amount of sodium sulphate was added. A diaphragm was employed and efficiencies of about 40 percent were obtained. Cowper-Coles (4) used a similar system in which the bath consisted of an acidified chromic chloride solution. Fereé (6) also used a highly acidified chromic chloride solution and obtained very pure metal. From concentrated solutions of chromic chloride and potassium chloride, he obtained good deposits on platinum with 45 percent efficiency. Neumann and Glaser (7) obtained steel-gray metal from solutions of the sulphate and chloride in a porous pot with efficiencies of about 50 percent. They found that elevated temperatures were detrimental to the character of the deposits. On carbon cathodes they obtained an efficiency of 85 percent. Chromic acetate was found to yield poor results. LeBlanc (8) was unable to prepare chromium by the methods of Placet and Bonnet, but with porous pots he obtained traces of metal. The baths that he found satisfactory can be regarded as acidified chrome alum solutions. Carveth and Mott (9) made a rather extensive research on deposition from solutions of chromic chloride and chromic sulphate in porous pots. They proved the importance of chromous ions in their baths. They found that high temperatures were detrimental to the character of the deposit and to the efficiency of deposition. They reported efficiencies varying from 10 to 50 percent, with good deposits at efficiencies around 30 percent. Dony-Henault (10) analyzed the

[1] The numbers in parentheses here and throughout the text refer to the bibliography at the end of the paper.

data of Carveth and Mott and concluded that the detrimental temperature effect reported was due to the formation of the green form [2] of chromic salts at elevated temperatures. He therefore predicted that successful deposits would depend on the chromic ion being in the violet form. This idea seems to have captured the imagination of electrochemists and has often been employed by them as a requisite of the desired solution. Voisin (13) was unsuccessful in repeating the work of Placet and Bonnet, but succeeded in duplicating Fereé's results. Sigrist, Winkler, and Wantz (17) stressed the importance of a diaphragm for the best results. From a chemical analysis of the deposits they concluded that the chromic acid bath is superior to baths of trivalent chromium.

Traube, Burmeister, and Stahn (18) worked with chromous solutions and obtained very impure dark deposits, but with high efficiencies. Pamfilov and Fillippuichev (24) used basic chromic sulphate solutions at room temperature and obtained fair results; the maximum efficiency found was 25 percent. Britton and Westcott (28) investigated sulphate and chloride solutions, both with and without diaphragms. They confirmed many of Carveth and Mott's results but failed to obtain their high efficiencies. An approximate analysis of the deposits showed them to be very impure. They recommended the use of a diaphragm and the addition of alkali salts, and emphasized the importance of the control of the hydrogen ion concentration. Fuseya and Sasaki (26) worked with trivalent chromium baths and reached the conclusion that for successful deposition the green form of the salts must be used and that the acidity must be regulated. It is noteworthy that they recommend the use of green rather than violet salts. Pamfilov and Federov (31) carried out experiments similar to those of Pamfilov and Fillippuichev but with plates instead of wires as cathodes, and obtained poor deposits and very low efficiencies. Britton and Westcott (32) followed up the ideas developed in their first research and investigated chromic acetate and complex tartrates and oxalates. The results were very unsatisfactory except with ammonium chromi-oxalate, which had been previously shown by Mazzuchelli (25) to give good deposits but with very low efficiencies. With complex citrate baths Yntema (33) obtained very good deposits. Liebreich (16) recommended the use of alkaline chromites.

As it is not certain that the investigators who got good plate really obtained deposits that were of commercial quality, it was considered advisable to investigate the problem from this point of view. Attention was therefore focused on the cathode processes, and in particular on the conditions for the best deposits. The above-cited literature indicates that both violet and green forms of chromic salts may be used, and that the chromous state is important. The solutions must be slightly acid and should preferably contain a large amount of alkali salt. For best results a diaphragm must be employed.

[2] For example, the violet chloride $[Cr(H_2O)_6]\ Cl_3$, is transformed by heating into the green compounds $[Cr\ (H_2O)_5Cl]\ Cl_2$ and $[Cr\ (H_2O)_4Cl_2]\ Cl$.

III. BATHS CONTAINING SIMPLE TRIVALENT SALTS

1. THEORY

The chemistry of trivalent chromium is complex and no one theory will explain the electrodeposition from all the possible compounds. The principal compounds existing in neutral and moderately acid aqueous solutions may be considered to fall into four classes.

Class I. The trivalent chromium exists as the violet hexa-aquo ion, $[Cr(H_2O)_6]^{+++}$, in conjunction with anions that do not exhibit a marked tendency to form molecules or molecular ions at room temperature. When the temperature is raised, an irreversible transformation occurs, which may be partial or complete. The resulting form may be a complex electrolyte (a positive molecular ion), an undissociated molecule, or a colloid. This behavior is known to occur with the chloride, bromide, and sulphate. The fluoride can be made to undergo the transformation but not by merely heating.

Class II. The violet hexa-aquo ion exists in conjunction with anions that do not exhibit any marked tendency for the formation of the green form even when the solution is heated. This class is exemplified by the nitrate,[3] perchlorate, fluosilicate,[4] and fluoborate.[5] Molecules and molecular ions containing the chromic ion and the negative radicles are absent.

Class III. The hexa-aquo ion is in equilibrium with a highly dissociated molecular ion. The only known member of this class is chromium dichromate.

Class IV. The trivalent chromium is bound strongly with the negative radicle to form undissociated molecules or negative molecular ions. The fluoride and sulphate give rise to this condition in special instances, but it is more frequently encountered with the organic anions such as acetate, oxalate, tartrate, and citrate.

The simple trivalent chromium salts are members of classes I and II. There has been a difference of opinion whether the violet or the green salt is preferable for electrodeposition from trivalent baths; both sides have brought forth apparently incontrovertible evidence. The majority of electrochemists assume that the violet form is necessary and that the difficulty of chromium plating is in keeping the salt violet. This idea owes its origin to Dony-Henault, whose original statement was, however, based upon false premises. He made this assumption to account for the fact that Carveth and Mott found that the baths operated at temperatures above 50° C. were inferior to those operated at lower temperatures. Dony-Henault pointed out that this is consistent with the fact that the green form is favored at high temperatures and the violet at low. It so happens that the puce [6] form of chromic chloride employed by Carveth and

[3] Strictly the nitrate is intermediate between classes I and II.

[4] According to the author's theory (34) of chromium deposition from the chromic acid bath, any anion of this second class is ineffective as an addition agent (as a substitute for sulphate). It would appear, therefore, that the proposed use (29), (30), of the fluosilicate in such a capacity is contrary to the theory. As emphasized in one of the patents (29) the fluosilicate merely acts as a source of fluoride, which latter is an effective addition agent. The instability of chromium fluosilicate was first established by Recoura (14). The apparent effectiveness of the fluosilicate ion as an addition agent to the chromic acid bath was first reported by Müller (20).

[5] That this ion should exhibit this characteristic is in accordance with the isomorphism of the perchlorate and fluoborate, as brought forth by Wilke-Dorfurt (19). It is interesting to note that the stable anions in this class are the ones that give the most satisfactory lead deposits, perhaps because they do not form molecules or molecular ions that include lead. This shows that complex ions are always not necessary for good deposits, though they are used in many plating baths for getting the best deposits.

[6] The lavender anhydrous chromic chloride whose solution can be effected only by employing chromous salts as catalysts.

Mott is known to give $[CrCl_2(H_2O)_4]Cl$ when dissolved. The sulphate which they employed was green. Most of the work has been done with the green salts, as they are the most common.

If the development of the proper bath (as has been suggested) merely depends on finding a salt that always remains violet, the solution of the problem would consist in selecting the most practical salt of class II. The electrochemically unstable nitrate and perchlorate [7] are ruled out. The fluosilicate is likewise eliminated for reasons previously mentioned. Accordingly, then, the logical salt to use would be the fluoborate.

In considering the principles of deposition from trivalent baths we shall assume a system in which only the simple, violet hexa-aquo chromic ion is present; the green form and the chromous ion are absent. The process of electrodeposition from such a system consists in effecting a reaction with a positive free energy change, the minimum value of which is given by the standard free-energy change. In actual electrolysis, the true value will be markedly different owing to polarization, but the relation of the free energy to that of another similar reaction of a different metal will not change appreciably. This amounts to saying that that portion of the polarization which is not dependent on the hydrogen overvoltage will be approximately the same for all systems considered. If this may be considered a close approximation in this case, it gives an illuminating picture of the results to be expected and which are actually found. Hence, if we compare the zinc and nickel baths with the case in hand, we should be able to show whether plating at high efficiencies is at all possible.

The energy of deposition of zinc is considerably higher than that of nickel. However, zinc possesses a high hydrogen overvoltage compared to that of nickel. Therefore, although zinc may not flash as well as nickel, once the cathode is covered with zinc the latter can be deposited with as high an efficiency as nickel. The situation with trivalent chromium is somewhat different. Its free energy of deposition is intermediate between that of nickel and zinc. Its hydrogen overvoltage [8] is even lower than that of nickel, hence, unlike zinc, it cannot be deposited with as high an efficiency as nickel with the same pH prevailing in the bulk of the solution. It is not possible to calculate the hydrogen-ion concentration of the cathode film from overvoltage data or from pH determinations in the bulk of the solution. There is little doubt, however, that in zinc and nickel deposition a hydrogen-ion concentration as low as 10^{-6} prevails. We shall assume conservatively that it is a 10^{-5}. It is then possible to show from the standard electrode potentials of chromium (-0.5v) and of nickel (-0.23v) that a hydrogen-ion concentration of not greater than 10^{-8} must exist in the cathode film before chromium can be deposited efficiently. This condition is impossible with an easily hydrolyzed ion like the violet chromic ion, which is completely precipitated at a hydrogen-ion concentration of about 10^{-5}.

The question arises as to just what differences are to be expected between the violet and green forms of class I, and as to whether the green compounds present a means of circumventing the difficulty.

[7] At the high current densities that must be employed in chromium deposition.

[8] No reliable values of the overvoltage on chromium have been reported, but experience in chromium plating indicates that it is considerably below nickel.

The usual pH determinations indicate (erroneously) that the green form is more readily hydrolyzed than the violet. Actually, however, the violet salts are more highly hydrolyzed than the green, which is demanded theoretically by the fact that the violet ion is more highly charged and has a smaller radius. The erroneous result is due to the formation of extraneous basic salts. If, as Bjerrum (11) has done, steps are taken to eliminate them or to use data that do not involve them, the hydrolysis constants fall in the expected order. The differences in free energy between the violet and the green forms have never been extensively measured, but the differences in heat content have been. Unfortunately this is a type of reaction in which Thomson's rule cannot be used to estimate the free energy change from the heat content change. However, as the two forms are coexistent and as equilibrium can be established between them, it follows that the free energy difference is small and of such a sign that the green form is favored at temperatures of 50° C and above; the violet is preferred at lower temperatures. This is in accordance with the few measurements of Demassieux and Heyrovsky (23). From a consideration of the hydrolysis constants, it might be concluded that with green solutions it should be possible to operate with a lower hydrogen-ion concentration than with the violet. However, it is an experimental fact that the point of precipitation of the hydroxide is roughly independent of whether the violet or green salts are used. This is caused by the fact (shown by Bjerrum) that the transition from the green to the violet form involves a basic salt as an intermediate step.

It follows from this analysis that no material advantage can be gained by working with the green salts. If, as will be shown later, the performance of the bath is largely dependent on the accumulation of chromous ions, the violet form should give better results than the green because, as Traube and Goodson (15) have shown, the efficiency of reducing trivalent chromium to the chromous state is much higher for the violet than for the green form. At room temperature the green form is more difficult to reduce than the violet form, even though it represents a higher (presumably more active) energy state. The explanation of this behavior lies in the modern interpretation of the process of electrolytic reduction (27). If we have two systems representing equal energy states, the reduction of the form that approaches the closer to the cathode occurs the more readily. At all events, there is no indication that baths having high efficiency are realizable with any members of class I or class II.

(2.) EXPERIMENTAL WORK

As stated, the primary aim was to see whether a trivalent bath could be developed which is comparable to the chromic-acid bath. The definition of the proper conditions for obtaining a bright deposit was largely empirical. Having once located the range of conditions of best deposits for a particular solution, the temperature, concentration, current density, and acidity were altered, in order to find the optimum conditions for obtaining a satisfactory deposit.

In order to test the common assumption that a permanently violet bath should give good deposits, a solution of chromic fluoborate, which had been predicted and shown to be a member of class II, was studied. Unlike most violet salts, the fluoborate, as expected from its

similarity to the perchlorate, is extremely soluble and there is little doubt that it is a strong electrolyte. The useful life of this bath (and of any trivalent chromium bath) was extremely short, because by its hypersensitivity to traces of heavy metals like copper.[9] Copper cathodes were generally used. The bath was about 1.8 M in trivalent chromium and contained free fluoboric acid, the concentration of which was varied to produce pH values from 1 to 3. No sulphate was present. It was established that bright chromium could be deposited with free acid, corresponding to a pH of 1.3 at 40° C. and 20 amp/dm^2. These deposits were "white", in comparison with the blue chromium obtained from the chromic-acid bath. The cathode efficiency of the best deposits was about 3 percent. There is little doubt that these conditions represent roughly the best performance of this bath. It is to be noted that the best deposits were obtained at rather high current densities. It was found that the plating range was exceedingly narrow, and that uniformly bright deposits could be obtained only when extreme precautions were taken to insure an even current distribution over the entire plate, which was done by shielding the edges. The oxidation of trivalent chromium was high at the magnetite anode, much higher than that found with the chrome-alum solution. There was nothing in the large number of experiments performed to indicate that the chromic fluoborate was markedly superior to other chromic salts such as potassium chromic sulphate.

More extensive experiments were made with potassium chromic sulphate solutions. Preliminary experiments showed that it was difficult to obtain good deposits from solutions of this salt alone. It was necessary to add an alkali salt (sodium sulphate), and free acid, which required the use of high current densities. As indicated in the introduction, operation at a low current density would be particularly desirable. For this reason, a rather extensive study was made of baths with a low acid content, but the quality of the deposit deteriorated as the acidity was reduced. Hence the operation of a trivalent chromium bath at low current density does not seem feasible. A Haring cell (21) was employed on these experiments to get uniform current distribution. The most satisfactory solution found contained 100 g/l of $KCr(SO_4)_2.12H_2O$, 300 g/l of Na_2SO_4, and 12 g/l of H_2SO_4. Bright deposits with a cathode efficiency of 6 percent were obtained at 30° C and at about 9 amp/dm^2. These deposits were almost of commercial quality,[10] but they were somewhat pitted and a perfect plate was never obtained. Even this result was difficult to obtain on account of the extremely narrow plating range. Variations of 5 percent from the optimum current density gave a completely dull deposit. The dull deposits were, however, fairly uniform in texture. The usual deposits obtained under the optimum conditions had a blotched appearance with dull and bright areas on various sections of the plate. A few uniformly bright deposits were obtained, but the unusually narrow plating range could not be widened by any variations.

As the development of a high-efficiency bath from simple trivalent salts is fundamentally unsound, further work in that direction was

[9] This fact would limit the commercial application to steel and nickel-plated objects. No other investigator seems to have called attention to this fact. It is possible to ruin the best bath by merely letting the copper cathode stand in the solution for a short time.

[10] Those deposited on a copper base left a dark residue on solution in acid, while those on nickel did not. This dark material is undoubtedly caused by the copper and not by any inherent property of the bath.

abandoned. The narrowness of the plating range can be readily explained if we assume that two processes are effective. It is only under extremely restricted conditions that two such widely divergent systems as chromic and chromous salts can be expected to yield a bright deposit. A variation of efficiency with time of operation of the bath was observed by Carveth and Mott and by Britton and Westcott (28). This effect was undoubtedly caused by the accumulation of chromous salts on continued electrolysis. It was not observed in the experiments reported here, in which no diaphragm was used; obviously such an effect will be small in the absence of a diaphragm, because the accumulation of a strong reducing agent like chromous ion would be impossible with an insoluble anode in the solution. If the presence of the chromous ion increases the efficiency, it might be desirable to use an entirely chromous solution.

IV. BATHS CONTAINING CHROMOUS SALTS

Chromous salts might be advantageous for two reasons, first, the energy required for deposition is lower, and second, the cathode film can reach a lower hydrogen ion concentration without precipitation of the hydroxide. Although no data exist on the hydrolysis of the chromous ion, it is likely that, as a bivalent ion, it hydrolyzes considerably less than a trivalent ion like the chromic. These two factors would assist in the attainment of a higher efficiency. However, it can be shown by reasoning similar to that already employed that there is no promise of an efficiency approaching that of nickel deposition.

Bunsen (2) obtained bright deposits from a chromous solution. Traube, Burmeister, and Stahn (18) got poor deposits from pure chromous solutions, but with very high efficiencies. Carveth and Mott (9) obtained high efficiencies, and stated that good deposits could be obtained with an efficiency of 30 percent. However, Britton and Westcott (28) failed to find such high efficiencies. The discrepancy can be readily explained by differences in technique. Carveth and Mott employed parchment separators whereas Britton and Westcott used ordinary clay ones. It is known that silica surfaces catalyze the oxidation of chromous ions by hydrogen ions. Further, Britton and Westcott stirred their solution, which is certainly not conducive to the accumulation of a large concentration of chromous ion. The use of concentric electrodes, which give good current distribution, leads to better results, as Pamfilov and Federov (31) have shown.

The first experiments were done with green chromic sulphate [11] in the cathode chamber, and with an auxiliary cathode of carbon.[12] An alundum porous pot was used, and the anode compartment contained either sodium sulphate or sulphuric acid or both. The entire apparatus was placed in a closed container through which carbon dioxide was passed continuously. The oxygen evolved at the anode was allowed to escape directly. The volume of liquid in the cathode compartment was three liters and that of the anode, 300 ml. With this relatively large apparatus, the conditions of plating were fairly constant. The initial concentration of chromic sulphate was three molar. Although no analysis was made to determine the degree of

[11] Chromic chloride could not be used (in the apparatus employed) on account of the free chlorine which forms at the anode and diffuses to the cathode where it prevents the accumulation of chromous ions.

[12] The usual lead cathode cannot be used on account of the poisoning action of the lead on the deposit.

reduction, this was certainly much higher than could be obtained with open vessels. The best results were obtained with neutral chromic sulphate in the cathode and sodium sulphate in the anode compartment. Even the best bright deposits had a dark sheen on them. The most striking feature was that the plating range was still extremely narrow. Bright deposits were obtained at 10 amp/dm^2 at 30° C. and the efficiency was 3 percent on the basis of bivalent chromium.

Solutions of pure chromous sulphate were prepared by dissolving electrolytic chromium in sulphuric acid in a closed apparatus with the air displaced by carbon dioxide. The first solution was 1.5 *M* in chromous sulphate and 0.05 *N* in free acid. A saturated solution of sodinm sulphate was used in the anode compartment. Again the bright plating range was found to be very narrow, in fact no entirely bright plate was produced. At 30° C., the best deposit was obtained at 30 amp/dm^2. The efficiency for a dull plate at a current density just above that for the bright plate was 12 percent for trivalent, or 8 percent for bivalent chromium; for the bright plate it must have been lower.

A more nearly neutral solution was obtained by adding insufficient acid to dissolve all the chromium. The salt concentration was approximately the same as in the previous case. From this solution no metal was deposited but merely brown chromous oxide.

Experiments under widely different conditions failed to reveal any wide plating range for chromous solutions, although high efficiencies were readily obtained for dull, impure deposits. It is at least probable that the reported high efficiencies for chromous or partially chromous solutions were obtained under conditions that gave commercially unsatisfactory deposits.

The surprising similarity in behavior between the chromous and chromic baths may be readily explained if the possible processes are considered. When conditions at the cathode permit the deposition of metallic chromium, the chromous ion is in a state where it can readily undergo exchanges of energy with the cathode; that is, it can be reduced by electrons from an external source. These same conditions permit the chromous ion to undergo any other chemical reaction that may be hindered in solution, but which may occur on the surface of the cathode. There are two such reactions, (*a*) the autoxidation and reduction of chromous ion ($3Cr^{++} \rightarrow Cr + 2Cr^{+++}$), and (*b*) the reaction between chromous ion and hydrogen ion ($Cr^{++} + H^{+} \rightarrow Cr^{+++} + \frac{1}{2}H_2$). Thermodynamic evidence shows that the first reaction is insignificant, but that the second is not, as it has a standard free energy change of -9 kg calories. It is known that the oxidation of the chromous ion by the hydrogen ion can be influenced by surface catalysts, particularly metals of a low hydrogen overvoltage. It has been shown (12) that the rate-determining step is the rate of diffusion of the solution to the metal. Since the cathode accelerates the frequency and ease of contact by virtue of its negative charge, and for the same reason work is put into the system, the reaction will occur very much more readily at a cathode than on an uncharged metal. It is hence impossible to obtain chromous ions on the surface of any metal, such as chromium, which possesses a low hydrogen overvoltage, without the production of a relatively large amount of chromic ions. Therefore the performance of the chromous bath will be similar to that of the chromic. The only essential difference is

that the chromous bath can be operated at higher current densities without the production of green oxide. This condition does not make an acid chromous bath promising. There are three ways for possible improvement: (*a*) to employ a base metal of a high hydrogen overvoltage, (*b*) to use very high current densities, and (*c*) to operate at a high pH. The first is automatically excluded by the properties of chromium, the second is undesirable, and the third is, as has been shown, impracticable.

V. BATHS CONTAINING COMPLEX CHROMIUM COMPOUNDS

Various possible methods of circumventing the difficulty that makes the operation of the simple trivalent chromium bath unfavorable as compared with the chromic acid bath have been discussed. Britton and Westcott (32) were quite aware of this difficulty, so they proposed to employ complex salts (members of class IV) of trivalent chromium which can exist in alkaline solutions without precipitating the hydroxide. This idea presents, according to Britton and Westcott, a valid means of circumventing the difficulty. However, the low metal ion concentration that makes the precipitation of the hydroxide difficult also makes the reduction of the trivalent chromium more difficult. This is an inescapable thermodynamic fact. Although no material improvement in efficiency can be expected, it may be that wider plating ranges can be obtained.

Britton and Westcott worked with chromium acetate, tartrate and oxalate. With ammonium chromi-oxalate they could obtain good deposits over a rather wide range but at very low efficiencies (less than 1 percent). The other solutions were worse than the simple salts. Mazzuchelli (25) has shown that such a bath would work, but at exceedingly low efficiency. There is a recent patent (22) which purports to describe a successful chromium chloride bath. This bath was apparently designed following the axiom of Dony-Henault. To obtain violet solutions the inventors added nitrate (which is likely to be reduced to ammonia) and oxalic acid. Ammonium salts were also added. Apparently the color of the chromi-oxalate was confused with that of the violet hexa-aquo ion. There is little doubt that the resulting bath reached a state in which it was principally the ammonium chromi-oxalate. The character of the deposit and the exceedingly low efficiency obtained in experiments on their baths confirm this conclusion.

Yntema (33) recommended the use of citrates. This work was repeated. The best deposits were lustrous, but dark, and the efficiencies were less than 1 percent.

VI. COMPARISON OF RESULTS WITH THE CHROMIC ACID BATH

A comparison of the trivalent chromium bath with the chromic acid bath will make clear why it seems improbable that a trivalent or bivalent bath can be designed which will be superior to the sexivalent bath. In a previous paper on the theory of chromium deposition (34), it was pointed out that the reduction of chromic acid to metal is an even more probable process thermodynamically than the liberation of hydrogen. That is, if it were possible to construct a reversible cell

which would be capable of yielding all possible degrees of reduction of chromic acid in their proper thermodynamic order, we would find chromium metal coming out before the liberation of hydrogen. With the trivalent state, the situation is the reverse, hydrogen would be liberated before the deposition of chromium. This argument is based on the plausible theory that in the chromic acid bath reduction occurs directly from the sexivalent state. A research is now in progress to confirm this hypothesis.

Further, it was proved that the deposition actually occurred with the cathode film quite acid (pH approximately 3). Thus we see that the deposition of chromium from a strong oxidizing agent presents a means of circumventing the difficulty that renders the trivalent bath impracticable. There is no doubt that trivalent chromium, under the conditions of acidity that obtain in chromium deposition from the chromic acid bath, would yield a current efficiency which would be considerably lower. That is only possible with a strong oxidizing agent. The difference between the chromic acid bath and the trivalent bath is exceedingly large when looked at from this point of view, and we do not see how any manipulation of the controllable factors can overcome this enormous difference.

If a superior chromium bath is to be made it will not lie in the direction of lower states of oxidation.

VII. CONCLUSIONS

(*a*) Simple trivalent chromium baths yield poor deposits at low efficiencies. The bright plating range is exceedingly narrow; (*b*) Chromous baths give results which are similar to those of the trivalent baths; (*c*) The use of complexes in order to solve the difficulties inherent in the trivalent bath is unsound; (*d*) The chromic acid bath is inherently superior to solutions containing lower valence compounds.

VIII. ACKNOWLEDGMENTS

The author is indebted to William Blum for suggestions and advice and to M. R. Thompson for measurements of pH.

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WASHINGTON, July 29, 1933.

U. S. Department of Commerce Bureau of Standards

RESEARCH PAPER RP605

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

THE UTILITY OF THE SPARK TEST AS APPLIED TO COMMERCIAL STEELS

By R. W. Buzzard

ABSTRACT

Typical spark streams of plain carbon steels, a variety of alloy steels including S.A.E. steels, nitrided steel and cast iron are described and illustrated. Definite spark stream characteristics were found in the steels studied for each of the alloying constituents, chromium, manganese, molybdenum, nickel, tungsten, and vanadium.

A supplementary study was made of the appearance of the metal pellets collected from the spark streams of the various types of steel. The presence in the steel of chromium, molybdenum, silicon, or vanadium in some cases imparts characteristics to the pellets which may aid in the identification of the steel.

The spark test may be depended upon as a means for classifying steels into groups of similar composition, but cannot be successfully used as a means of identifying an unknown steel.

On the basis of the data given by the combined spark test and pellet test a sorting chart has been prepared. This chart indicates the possibility of sorting by the spark and pellet tests over 2,100 combinations of two steels of a total of 2,500 possible combinations of any two steels in the chart.

CONTENTS

	Page
I. Introduction	528
II. Method	528
1. Equipment	528
2. Photography	528
3. Nomenclature of spark trajectory	528
4. Theory of spark stream	529
5. Method of study	529
III. Materials and results	530
1. Plain carbon steels	530
2. Nickel steels	531
3. Chromium steels	532
4. Nickel-chromium steels	533
5. Chromium-vanadium steels	534
6. Molybdenum steels	534
7. Tungsten steels	535
8. Silicon-manganese steel	536
9. Manganese steels	536
10. Cast iron	537
11. Nitrided steel	537
12. Miscellaneous alloys	537
13. Pellet test	538
IV. Summary	539
1. Sorting chart for S.A.E. steels	539
2. Conclusions	539
V. Acknowledgments	540

I. INTRODUCTION

The difference in the sparks produced by applying steels of different compositions against a revolving abrasive wheel was first described in 1804.[1] Relatively little detailed information, however, has since been published. The published records which have appeared, with two exceptions,[2 3] have used written descriptions or drawings to record the results of the study of this phenomenon. It is the purpose of this paper, which summarizes the results of the application of this method of testing to a wide variety of steels, to emphasize points often overlooked in the use of the spark test, to present photographs of representative spark streams,[4] as well as to discuss some new aspects of the subject.

The spark test is limited to two fields of application; namely, (1) for inspection and, (2) for sorting of unknown steels from a lot of steel of known chemical composition. The test cannot be depended upon for identifying steels of unknown composition; such determinations can be made only by chemical analysis.

II. METHOD

1. EQUIPMENT

The equipment required is relatively simple. A ⅛-horsepower motor, weighing about 8 pounds, operating at a shaft speed of 15,000 rpm was used in this work. An abrasive wheel of alundum (41S) 1¼ inches in diameter and ⅜-inch thick was mounted on the shaft. The working peripheral speed of the wheel was about 5,000 fpm.

Colored glasses should be worn, as constant observation of spark streams with the unprotected eye is harmful. The color of the glass is not so important. It must be of sufficient density to relieve eye strain, but as light as possible so it will not obscure the characteristics of the spark stream.

2. PHOTOGRAPHY

Either a motion-picture camera or an ordinary camera for single exposures can be used successfully for such photographs as are reproduced in this paper. Since a good photograph was more a matter of chance than of skill, a motion-picture camera was regularly used so that more individual records could be made with greater ease and speed, and thus give a larger number of photographic records from which to choose. It was necessary to use a fast lens with a fast panchromatic film. Extremely rapid exposures were out of the question as it was desired to record the entire trajectory of some of the particles. Short time-exposures were made against a dark background to allow the stream to expose itself.

3. NOMENCLATURE OF SPARK TRAJECTORY

In describing spark streams various investigators have used different terms. To make it easier to follow this description, the outstanding characteristics have been indicated on typical photographs as follows:

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[1] de Manson, Jacques, Traité du Fer et de l'Acier, Paris, 290 pp., 1804.
[2] E. Pitois and J. D. Gat, Sparking of Steel. Chemical Publishing Co., 1929.
[3] W. G. Hildorf and C. H. McCollam, Classifying Steels by Sparking, Metal Progress, Feb. 1933. (The author refers the reader to this paper for recommended practice for the routine application of the spark test.)
[4] Photographs for record only, all routine spark testing is by visual examination.

The general spark stream characteristics typical of carbon in steel as shown in figure 4, the "flower burst" in figure 10, the "spearpoint" in figure 21 and the "jacketing effect" in figure 25.

The plain carbon steels were studied in groups classified on the basis of similar spark streams. These groups are designated as brackets. Three such brackets were used; the lower carbon bracket including open hearth iron, and carbon steels through S.A.E. 1045 (Society of Automotive Engineers); the medium carbon bracket, steels from S.A.E. 1045 through S.A.E 1070; and the high carbon bracket, the high carbon steel, S.A.E. 1070, and above.

4. THEORY OF SPARK STREAM

The characteristic appearance of the spark stream is apparently to be attributed chiefly to the oxidation of the carbon in the steel. Alloying elements may contribute minor characteristics to the stream. It appears probable that as the grinding wheel tears off small particles of steel the work done causes the temperature to rise. This rise in temperature may also be increased by a "pyrophoric" oxidation effect resulting from the rapid surface oxidation of small particles which are torn away from the steel specimen with perfectly clean oxide-free surface. The particles of steel are heated to such a degree that they become fused, at least superficially, and tend to become spherical. Oxygen and carbon react in the heated portions of the particles to produce CO_2 and perhaps CO.

The oxide scale formed on a plain carbon steel is not very tenacious and easily flakes off. The gas which forms within the heated spherical particles escapes through this easily fractured skin and gives rise to the "series spark bursts." A comparatively smooth pellet which shows a slight pattern on the surface remains after the particle cools. Alloying elements in the steel may change the characteristics of the oxide film in such a manner as to give the various spark characteristics.

The entire spark stream consists of the trajectories of a multitude of glowing particles with the accompanying "bursts." As the carbon content of the steel is increased, the number and intensity of the "carbon bursts" increase.

5. METHOD OF STUDY

The study of the spark stream is simplified if the trajectory is considered to be divided into four sectors as shown in figure 4. The first sector is located directly adjacent to the abrasive wheel. A characteristic color, not necessarily the same as the spark stream color, is sometimes observed here, and the spark of nickel can often be detected in this portion. The second sector covers the dense portion of the stream between the first sector and the carbon bursts. The only pronounced characteristic observed in this sector is that of color. The third sector includes the well-formed carbon bursts at the end of the stream and it is in this sector that the characteristics imparted to the spark by the alloying constituents chromium, molybdenum, manganese, carbon, sulphur, nickel, vanadium, tungsten, and silicon are best observed. The invisible portion of the trajectory following the "bursts" may, for sake of completeness, be considered as the fourth sector although it is not really a part of the spark. The study of the "burnt out" particles or pellets col-

ected from this fourth sector gives some information concerning steels containing chromium, vanadium, molybdenum, or high alloy content in general.

III. MATERIALS AND RESULTS

A large variety of steels were examined. These consisted of representative steels of the various classes in the S.A.E. series, together with many others donated by different manufacturers. In the accompanying tables are listed the compositions of the steels which gave the typical spark streams illustrated in the figures of this paper. Steels of many additional compositions were studied in each group. The entire range of chemical compositions of steels upon which was based the selection of typical spark stream photographs is indicated in the following sections which summarize the observations made on the different materials. In these observations, a distinction has been made between two kinds of "carbon bursts." This distinction can readily be made and the characteristics of two types are indicated by the designations used, namely, the "series" bursts, and the "flower" bursts.

TABLE 1.—*Composition of plain carbon steels studied*

Designation	C	Mn	P	S	Si	Ni	Cr	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
Open hearth iron	0.01	0.003	0.02	0.020	0.01	0.02	0.01	1, 2
S.A.E. 1010	.09	.30	.011	.026	.02	.12	.02	1
S.A.E. 1015	.16	.51	.017	.026	.15	.20	.06	1
S.A.E. 1020	.20	.43	.006	.041	------	.008	.008	1
S.A.E. 1030	.32	.53	.01	.036	.064	------	------	3, 4
S.A.E. 1045	.44	.68	.016	.025	.23	.27	.06	3
S.A.E. 1050	.46	.57	.036	.042	.20	------	------	3
S.A.E. 1070 [1]	.70	.22	.023	.011	.23	------	.03	3
S.A.E. 1080 [1]	.76	.50	.028	.026	.28	.23	.28	5
S.A.E. 10100 [1]	1.06	.24	.014	.016	.18	------	------	5
S.A.E. 10120 [1]	1.22	.22	.015	.016	.25	------	------	5
S.A.E. 10130 [1]	1.29	.23	.017	.017	.23	------	.05	5

[1] Designated in accordance with S.A.E. numbering system for plain carbon steels; not official S.A.E. steels.

1. PLAIN CARBON STEELS

In table 1 are listed the compositions of the plain carbon steels which produced the representative spark streams shown in figures 1, 3, and 5. Six plain carbon steels in addition to those of table 1 were studied. These steels, all of carbon contents within the range of 0.23 to 1.12 percent, revealed no additional spark stream characteristics. In figure 4, the notations define the various terms used in describing the sparks. The simplest stream observed was that of open hearth iron. The stream was full, but on account of the absence of carbon not very brilliant. In general, the stream was dull red and the burst consisted simply of a swelling of the main line or carrier with occasional sprigs at the base of the enlargement. In steels containing as little as 0.10 percent carbon, the carbon burst was distinctly visible, usually as a terminal forked tongue. With the appearance of the forked tongue the stream brightened throughout.

The true "series burst" typical of this type of steel first appeared in steels containing approximately 0.15 percent carbon. The simplest

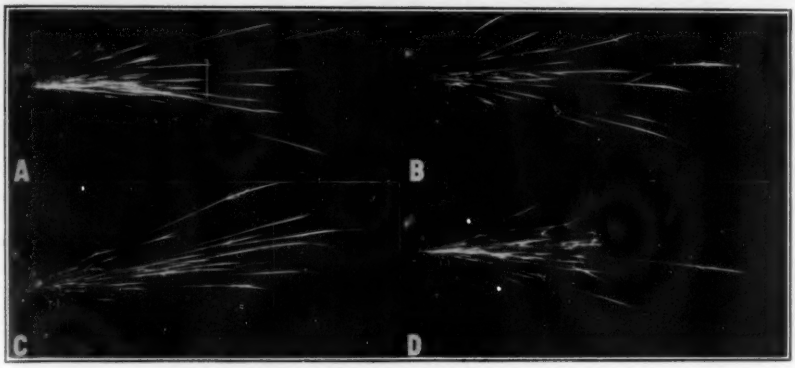

FIGURE 1.—*Typical features of spark streams of low-carbon steel.*

A, Open-hearth iron, single sprig bursts, dark tips; B, S.A.E. 1010 steel, two or three ray bursts, dark tips; C, S.A.E. 1015 steel, series bursts of sprigs; D, S.A.E. 1020 steel, series burst of sprigs and rays, buds.

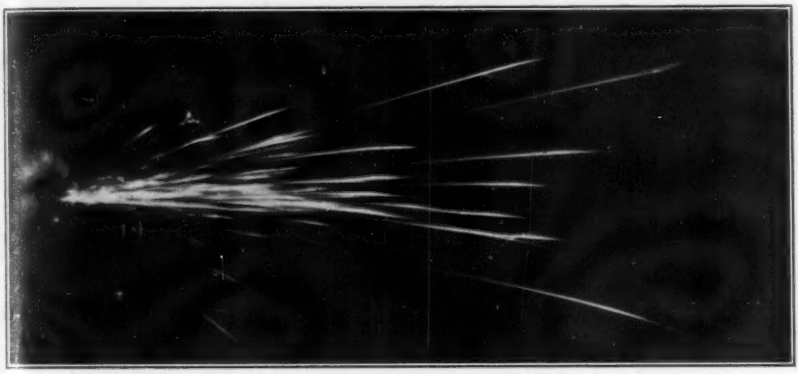

FIGURE 2.—*Spark stream of open-hearth iron. Enlargement of 1A.*

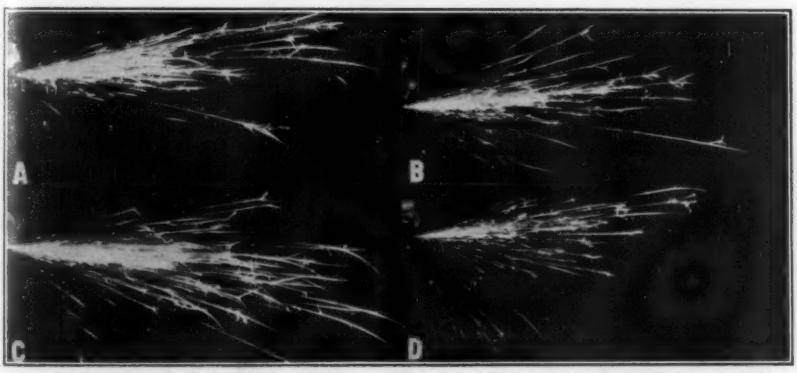

FIGURE 3.—*Typical features of spark streams of medium-carbon steel.*

A, S.A.E. 1030 steel, long tongues, series burst, buds and stars; B, S.A.E. 1045 steel, bright stream, series burst having preliminary bursts, buds, stars of the secondary type; C, S.A.E. 1050 steel, darkened sprigs and secondary carriers; D, S.A.E. 1070 steel, high carbon burst, finer carrier, shorter tongue. No sprigs and few rays. Full development of all carbon characteristics.

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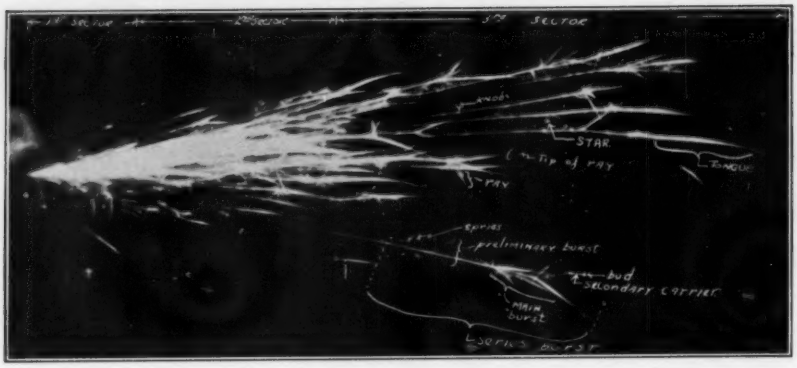

FIGURE 4.—*Spark stream of S.A.E. 1030 steel showing the various characteristics of the carbon burst.*

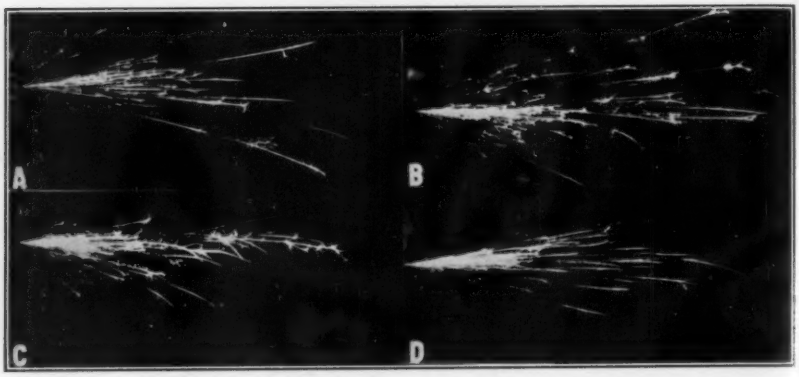

FIGURE 5.—*Typical features of spark streams of high-carbon steel.*

A, S.A.E. 1080; B, S.A.E. 10100; C, S.A.E. 10120; D, S.A.E. 10130: Shows development of all carbon characteristics and streams darker in general.

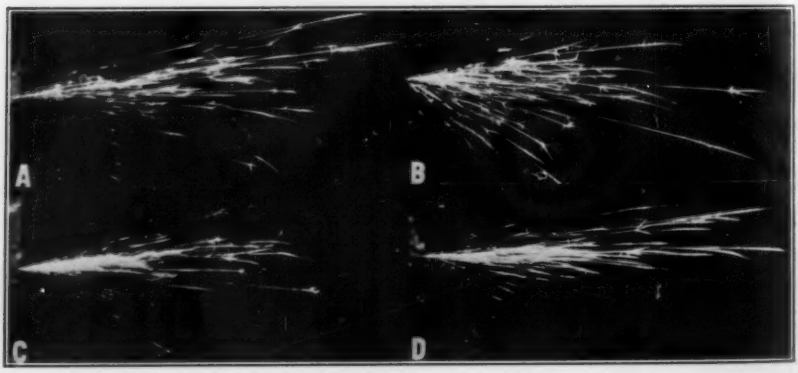

FIGURE 6.—*Typical features of spark streams of S.A.E. nickel steels.*

A, S.A.E. 2315 steel, forked tongues, slightly suppressed and compact burst; B, S.A.E. 2335 steel, forked tongues, slightly suppressed and compact burst; C, S.A.E. 2350 steel, forked tongues, slightly suppressed and compact burst; D, S.A.E. 2512 steel, forked tongue bursts, nickel spark.

form of the series burst consisted of a number of simple sprigs. With increase in carbon content above 0.15 percent, the length of the series burst was increased and buds and then stars appeared, all of which increased in number as the carbon content was increased. The maximum tongue length was obtained in steel containing about 0.30 percent carbon, and the maximum length of burst at 0.45 percent carbon. This was also the most spectacular stream observed.

The entire spark stream lightened in color as the carbon content was increased up to 0.45 percent and then gradually darkened in the steels of progressively higher carbon content, this being very pronounced in steel containing 1.00 percent carbon or more. In testing all of the steels of the high carbon class, a marked tendency for the spark to be carried entirely around the periphery of the abrasive wheel was noted.

2. NICKEL STEELS

The nickel steels listed in table 2, and in addition those corresponding to S.A.E. Nos. 2320, 2330, 2340 and 2345, were studied. Photographs representative of the spark streams are given in figures 6, 7, and 8.

TABLE 2.—*Composition of nickel steels studied*

Designation	C	Mn	P	S	Si	Ni	Cr	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
S.A.E. 2315	0.16	0.68	0.014	0.03	0.26	3.45	0.09	6
S.A.E. 2335	.36	.64	.018	.031	.24	3.41	------	6–7
S.A.E. 2350	.47	.65	.021	.025	.25	3.45	.06	6
S.A.E. 2512	.13	.45	.019	.023	.22	5.03	.16	6
N 1	.39	.55	.016	.032	.17	1.25	.62	8
N 2	.29	.74	.025	.017	.17	3.52	.14	8
N 3	.08	.31	.020	.016	.10	5.12	.15	8
N 4	.14	1.70	------	------	.16	47.40	------	------
	.05	.21	------	------	.31	34.27	------	8

Nickel was found to impart to the spark stream two characteristics which are useful in the sorting of a lot of mixed steels of known composition. The first, the "nickel spark", designated here as a "jacket", can be observed in either the first sector on the short carriers, or in the third sector at the base of the well-developed carbon bursts, but it is probably the most difficult alloy characteristic to observe. The second characteristic is the split tongue observed within many of the bursts instead of the usual single tongue.

The characteristics of the nickel spark can probably be attributed to the fact that the surface oxide layer on a particle of nickel steel is more tenacious than that on a plain carbon steel. It may follow that the formation of this layer on a nickel steel is so rapid and the film so tenacious that it is distended by the pressure developed within the molten pellet. Thus, the molten sphere may enlarge before bursting and at the high rate of speed of travel will produce a constantly broadening streak of light in its path and result in the oval flare at the base of the burst known as the nickel spark.

In the steels of the S.A.E. series, containing 3.00 to 3.60 percent nickel, the color of the stream was slightly darker than, but otherwise similar to, that of the spark stream of the corresponding plain carbon

steels. The bursts were compact and the tongues slightly shorter, the split tongue effect being a common occurrence. The characteristic nickel spark was readily observed in the steels low in carbon (0.15 percent) but became progressively fainter with increasing carbon content until, at 0.35 percent, it was practically unobservable. In general, the spark stream of a steel containing 5 percent nickel and 0.15 percent carbon was very similar to that of the corresponding plain carbon steel. The principal differences were the slightly suppressed burst, the darkening of the stream, and the nickel spark.

All of the alloy steels of high nickel content produced spark streams. An alloy of the invar type containing 34.27 percent nickel gave a stream of yellow streaks, whereas one containing 47.4 percent nickel produced a stream too dark to permit photographing it. A specimen of commercial nickel gave a spark stream, but one of monel metal gave none.

3. CHROMIUM STEELS

Table 3 gives the compositions of the chromium steels which produced the representative spark streams illustrated in figures 9, 11, and 12. The spark streams of some 20 chromium steels were studied in addition to those listed in table 3. The composition of these steels, however, are all closely approximated by the selected steels of table 3.

TABLE 3.—*Composition of chromium steels studied*

Designation	C	Mn	S	P	Si	Ni	Cr	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
S.A.E. 5120	0.21	0.70	0.018	0.029	0.18	0.19	0.73	9
S.A.E. 5130 [1]	.32	.63	.021	.019	.33	.....	1.06	.....
S.A.E. 5140	.45	.61	.023	.018	.24	.....	.91	9-10
S.A.E. 5150	.51	.72	.031	.024	.23	.....	.86	9
S.A.E. 52100	1.02	.37	.027	.014	.42	.....	1.40	9
C 3	.39	.64	.040	.026	.42	.....	.80	11
C 7	.92	.33	.027	.015	.31	.....	1.68	11
C 10	.72	.49	.025	.008	.....	.....	2.18	11
C 11	.90	.35	.....	.....	.22	.....	3.40	11
C 12	.71	.34	.....	.....	.37	.....	4.88	12
C 13	.10	.41	.004	.019	.33	.....	11.50	12
C 17	2.25	.60	.....	.....	.....	.....	13.00	11
C 18	.34	.34	.017	.024	.29	.30	13.00	11
C 23	.35	.25	.029	.016	.22	.28	14.36	11
C 25	.10	.40	.03	.03	.50	.....	17.00	11
C 26	1.00	.....	.....	.....	.....	.....	17.00	11
C 30	.25	.50	.03	.03	.75	.....	25.00	11

[1] Designated in accordance with S.A.E. numbering system for chromium steels; not an official S.A.E. steel.

The spark stream of chromium steels differed from that of plain carbon steels in that the carrier lines were finer and slightly darker. The bursts were of the flower type. These characteristics may be assumed to be associated with a strong oxide film which forms over the surface of the particles. The particle shatters under high internal pressure and a flowerlike burst results.

In the S.A.E. chromium steels whose carbon content did not exceed 0.45 percent, the spark stream resembled that of the plain carbon steels with the outstanding exception of the flower-like burst. In steels containing more than 0.45 percent carbon, the stream very closely resembled that of the corresponding plain carbon steel with the exception that the carrier lines were finer and somewhat darker.

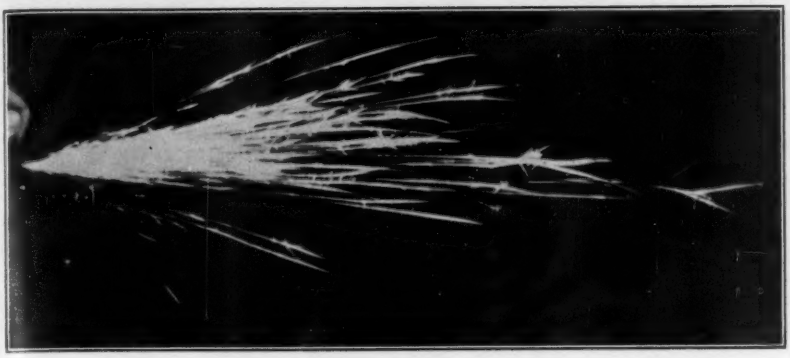

FIGURE 7.—*Spark stream typical of nickel steel showing the nickel spark and the forked tongue effect. Type S.A.E. 2335 c.f. (fig. 4).*

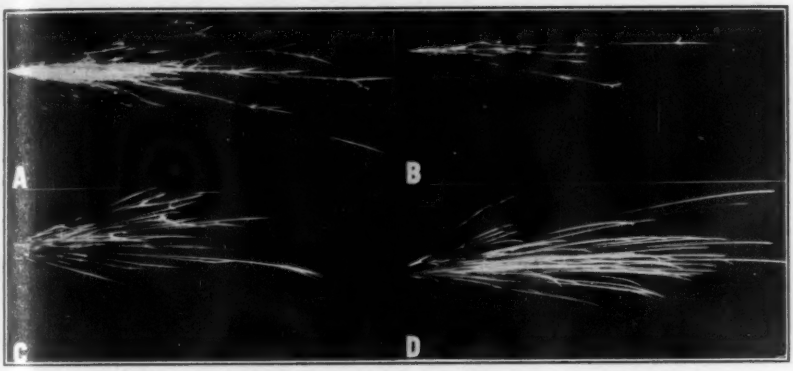

FIGURE 8.—*Typical features of spark streams of special nickel steels.*

A, Steel N1 (0.39 C, 0.62 Cr, 1.25 Ni), forked tongues; B, steel N2 (0.29 C, 3.52 Ni), nickel spark; C, steel N3 (0.08 C, 5.12 Ni), carbon burst absent, dark tips; D, invar, dark-red blunt carriers.

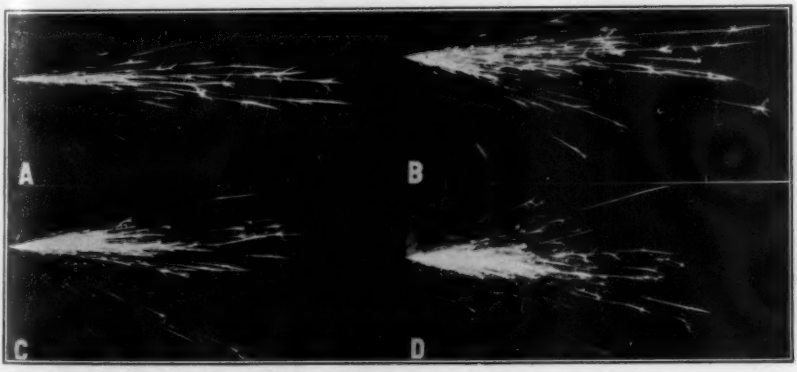

FIGURE 9.—*Typical features of spark streams of S.A.E. chromium steels.*

A, S.A.E. 5120 steel, flower bursts, slightly darker color; B, S.A.E. 5140 steel, flower bursts, more rays in burst; C, S.A.E. 5150 steel, flower bursts, more rays in bursts, fine carriers; D, S.A.E. 52100 steel, high carbon burst, fine carriers, slightly dark in color.

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FIGURE 10.—*Spark stream of typical chromium steel showing the flower burst. Type S.A.E. 5140, c.f. (fig. 4).*

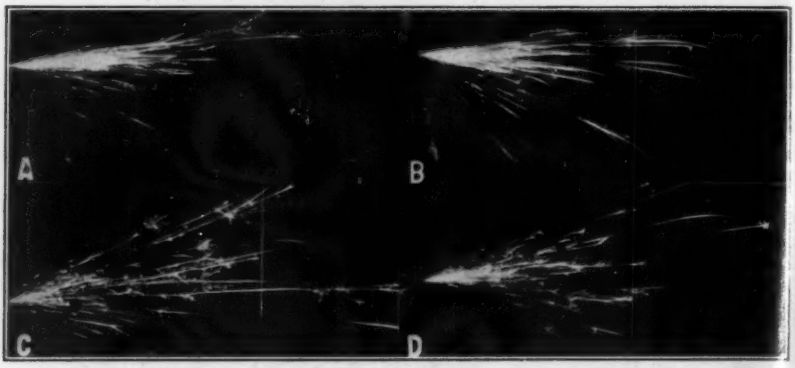

FIGURE 11.—*Typical features of spark streams of low-chromium steels.*

A, Steel C3 (0.39 C, 0.30 Cr), flower burst; B, steel C7 (0.92 C, 1.68 Cr), compact high carbon burst; C, steel C10 (0.72 C, 2.18 Cr), compact high carbon burst, fine carriers; D, steel C11 (0.90 C, 3.40 Cr), compact high carbon burst, fine carriers.

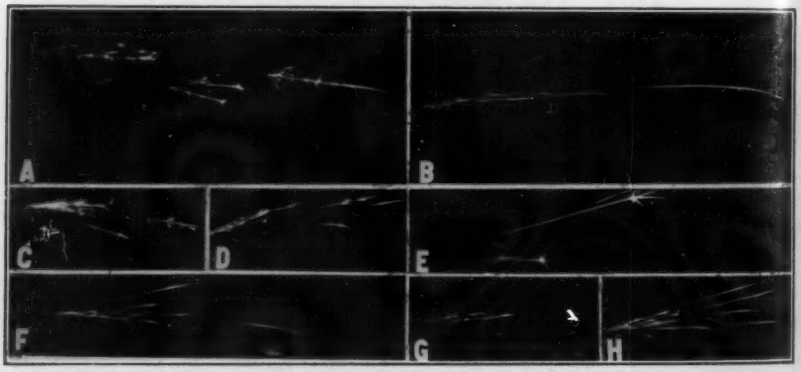

FIGURE 12.—*Typical features of spark streams of high-chromium steel.*

A, Steel C12 (0.71 C, 4.80 Cr), scant stream, typical carbon burst; B, steel C13 (0.10 C, 11.50 Cr), scant stream, disjointed red lines, suppressed carbon burst; C, steel C17 (2.25 C, 13.00 Cr), scant, short stream, occasional typical carbon burst; D, steel C18 (0.34 C, 13.00 Cr), scant, short stream, suppressed carbon burst; E, steel C23 (0.35 C, 14.36 Cr), scant, short stream, suppressed carbon burst; F, steel C25 (0.10 C, 17.00 Cr), scant, short stream, suppressed carbon burst; G, steel C26 (1.01 C, 17.00 Cr), scant, very short stream, carbon burst suppressed; H, steel C30 (0.25 C, 25.00 Cr), scant, very short stream, carbon burst suppressed.

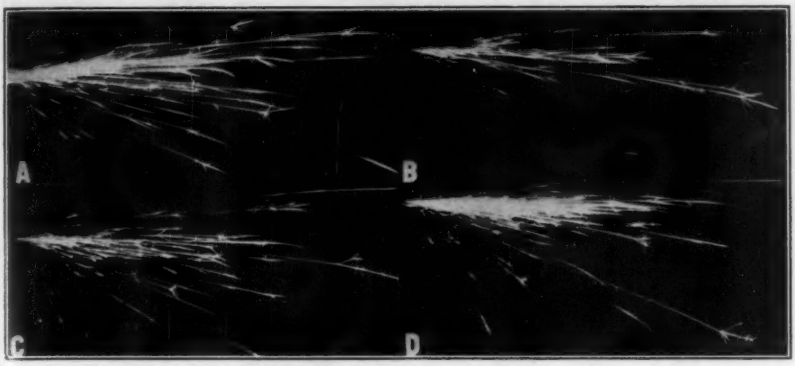

FIGURE 13.—*Typical features of spark streams of S.A.E. chromium-nickel steels.*

A, S.A.E. 3115 steel, nickel spark, flower burst, c.f. (fig. 1C); B, S.A.E. 3120 steel, flower burst, c.f. (fig. 1D); C, S.A.E. 3130 steel, flower burst, c.f. (fig. 3A); D, S.A.E. 3140 steel, flower burst, c.f. (fig. 3B).

FIGURE 14.—*Spark stream typical of chromium-nickel steel. Type S.A.E. 3130, c.f. (fig. 4).*

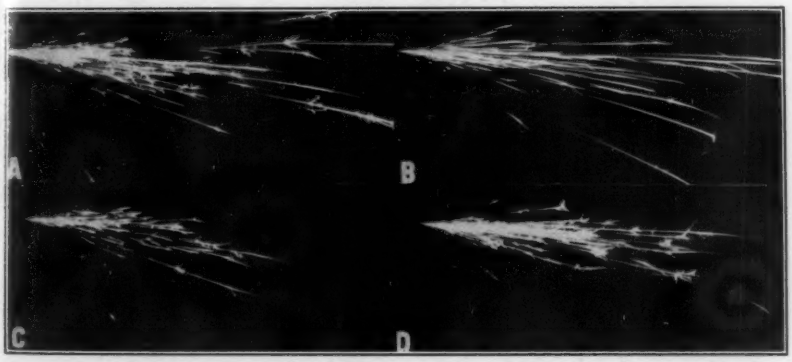

FIGURE 15.—*Typical feature of spark streams of S.A.E. chromium-nickel steels.*

A, S.A.E. 3240 steel, flower bursts; B, S.A.E. 3312 steel, small bursts, nickel spark, dark stream; C, S.A.E. 3340 steel, flower burst, dark stream; D, S.A.E. 3435 steel, dark streams, flower burst.

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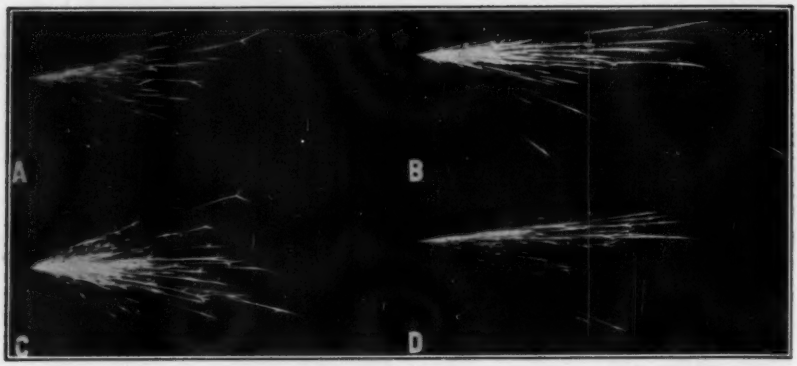

FIGURE 16.—*Typical features of spark stream of special chromium-nickel steels.*

A, Steel NC1 (0.19 C, 1.35 Ni, 0.51 Cr), flower bursts; B, steel NC4 (0.48 C, 1.65 Ni, 1.04 Cr); C, steel NC5 (0.28 C, 2.50 Ni, 2.50 Cr), flower burst; D, steel NC7 (0.24 C, 5.80 Mn, 10.04 Ni, 1.78 Cr), flower burst, jackets.

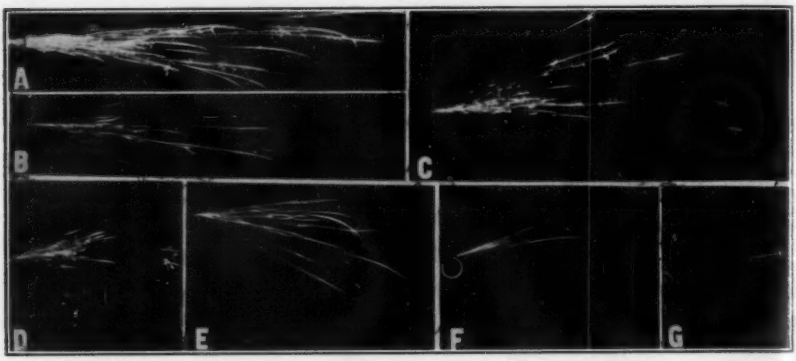

FIGURE 17.—*Typical features of spark streams of special chromium-nickel and chromium-vanadium steels.*

A, Steel V1 (0.45 C, 0.90 Cr, 0.20–0.25 V), carbon burst; B, steel V4 (0.40 C, 13.00 Cr, 1.00 V), no spearpoints, suppressed carbon burst; C, steel V2 (0.94 C, 1.44 Cr, 0.20 V), carbon burst; D, steel V3 (2.20 C, 12.00 Cr, 0.75 V), short scant stream; E, steel NC8 (0.15 C, 11.80 Ni, 13.00 Cr), banded wheel, scant stream, red disjointed lines, spearpoints; F, steel NC16 (0.10 C, 8.26 Ni, 18.82 Cr), banded wheel, scant stream, red disjointed lines, some spearpoints; G, steel NC20 (0.15 C, 11.00 Ni, 21.00 Cr), banded wheel, single carrier stream.

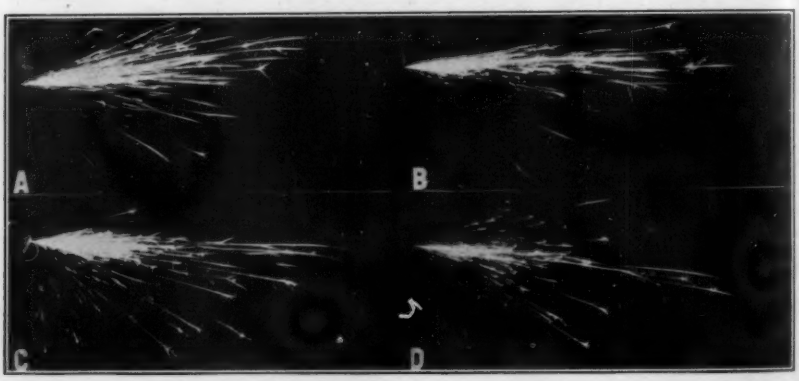

FIGURE 18.—*Typical features of spark streams of S.A.E. chromium-vanadium steels.*

A, S.A.E. 6120 steel, flower bursts, occasional spearpoint; B, S.A.E. 6140 steel, flower burst; C, S.A.E., 6150 steel, carbon burst; D, S.A.E. 6195 steel, carbon burst.

Only by very close examination could the flower bursts, which seem to be characteristic of chromium steels of lower carbon content, be detected in the spark stream.

Many of the special alloys studied (table 3) had high carbon content. All the steels (except C 3) containing less than 5 percent chromium contained at least 0.70 percent carbon. The spark streams produced were all typical of the carbon contents. The steels of highest chromium content were of the stainless type. A steel of this kind with a low carbon content gave a very short spark stream in which tongues showed a tendency to be tipped with spearpoints as is shown in the photographs. The periphery of the rotating abrasive wheel was brightly banded. A short, dark spark stream was obtained with the high chromium steels of higher carbon content. Although the stream was greatly suppressed, the carbon bursts were full and typical of the high carbon content. In the steels of highest chromium content (15 to 30 percent chromium) the spark stream was inconspicuous, and was entirely lacking in the steel containing about 30 percent chromium or more.

TABLE 4.—*Composition of nickel-chromium steels studied*

Designation	C	Mn	P	S	Si	Ni	Cr	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
S.A.E. 3115	0.15	0.46	0.017	0.024	0.20	1.31	0.60	13
S.A.E. 3120	.19	.47	.020	.029	.18	1.26	.62	13
S.A.E. 3130	.32	.46	.019	.031	.23	1.12	.63	13–14
S.A.E. 3140	.39	.62	.033	.036	.26	1.18	.62	13
S.A.E. 3240	.41	.44	.028	.022	.26	1.73	1.10	15
S.A.E. 3312	.10	.44	.019	.023	.22	4.14	1.49	15
S.A.E. 3340	.40	.50	.021	.021	.23	3.39	1.56	15
S.A.E. 3435	.36	.45	.028	.023	.22	4.14	1.49	15
NC 1	.19	.52	.012	.024	.15	1.35	.51	16
NC 4	.48	.46	.015	.009	.18	1.65	1.04	16
NC 5	.28	------	------	------	------	2.50	2.50	16
NC 7	.24	5.80	------	------	------	10.04	1.78	16
NC 8	.15	.50	------	------	.45	11.80	13.00	17
NC 15	.14	.70	.009	.017	2.02	8.42	18.06	
NC 16	.10	.34	.019	.023	.49	8.26	18.82	17
NC 20	.15	.60	------	------	.95	11.00	21.00	17

4. NICKEL-CHROMIUM STEELS

In table 4 are the compositions of the nickel-chromium steels whose spark streams are illustrated in figures 13 to 17 inclusive. The spark streams shown (figs. 13 and 15) are typical of the entire group of simple nickel-chromium steels examined. Steels of many compositions in addition to those listed in table 4 were studied and their spark streams compared with those selected as typical. The additional compositions included S.A.E. 3125, 3135, 3235, and 3250 steels as well as 24 special nickel-chromium steels. The carbon content of these special steels ranged from 0.8 to 2 percent. The maximum alloy content was as follows: Manganese, 1.28 percent; nickel, 35 percent; chromium, 25 percent; and silicon, 2.8 percent. One composition contained 0.2 percent molybdenum; another, 1.7 percent vanadium; and a third, 1.35 percent copper.

The combination of the two alloying elements, nickel and chromium, resulted in a suppression of the nickel effects and a strengthening of the chromium effects. In the S.A.E. steels the spark streams were similar to those of the corresponding carbon chromium steels

already discussed, though slightly darker in color. In the low-carbon steels of this type the nickel spark was detected but not in steels containing 0.30 percent carbon or more. As the combined nickel-chromium content was increased, a tendency for the stream to darken was observed.

Spark streams typical of a group of special alloy steels (NC 1 to NC 20, table 4) are shown in figures 16 and 17. It was found that steels of low nickel-chromium content produced spark streams in which the bursts were well developed. In steels of higher nickel-chromium content, 12 percent or more chromium, the carriers were darkened and the stream scanty. Only a few bright carriers, many of which were tipped with a spear point, were present in the spark stream, and it consisted largely of dull red streaks. In the steels high in both carbon and nickel-chromium content the stream was short, scanty, and dark, with bursts characteristic of the high carbon content. As in the high chromium steels, the periphery of the wheel was brightly banded.

In general, as the nickel-chromium content increased the character of the spark stream changed from bright carrier lines to dull red, disjointed lines. A high nickel-chromium content tended to suppress the stream entirely. This result was obtained when the combined percentages of nickel and chromium totaled approximately 30 percent. Steel NC 7, relatively high in manganese, gave a bright stream; evidently the manganese counteracted the effect of the chromium. Steel NC 15, containing 2 percent silicon, gave a stream of disjointed red lines.

TABLE 5.—*Composition of chromium-vanadium steels studied*

Designation	C	Mn	P	S	Si	Ni	Cr	V	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
S.A.E. 6120	0.21	0.70	0.019	0.034	0.45	0.18	0.73	0.13	18
S.A.E. 6140	.45	.66	.020	.011	.23	----------	.88	.16	18
S.A.E. 6150	.50	.50	.040	.040	----------	----------	.90	.17	18
S.A.E. 6195	1.06	.34	.017	.022	.27	.12	1.44	.17	18
V 1	.45–.50	.45–.55	.025	.025	.25–.35	----------	.90	.20–.25	17
V 2	.94	.34	.017	.009	.26	.09	1.44	.20	17
V 3	2.20	----------	----------	----------	----------	----------	12.00	.75	17
V 4	.40	----------	----------	----------	----------	----------	13.00	1.00	17

5. CHROMIUM-VANADIUM STEELS

The spark streams of all the S.A.E. steels (including 6125, 6130, 6135, and 6145 not listed in table 5 and the special steels studied (table 5)) were characterized by the chromium content rather than the vanadium (figs. 17, 18, and 19). Vanadium as an alloying element caused spear points to appear in the stream at the terminals of some of the carrier lines. This phenomenon, however, could not be depended upon as a reliable means of identification, although it is useful as an aid.

6. MOLYBDENUM STEELS

In addition to the molybdenum steels listed in table 6, spark streams were studied of compositions corresponding to S.A.E. steel 4140, and the so-called "4130X", as well as of seven special molybdenum steels containing 0.15 to 0.40 percent carbon, 0.15 to 3.6 percent nickel, 0.04 to 0.9 percent chromium, and 0.25 to 0.45 percent molybdenum.

FIGURE 19.—*Spark stream typical of chromium-vanadium steel. Type S.A.E. 6130 steel, c.f. (fig. 4).*

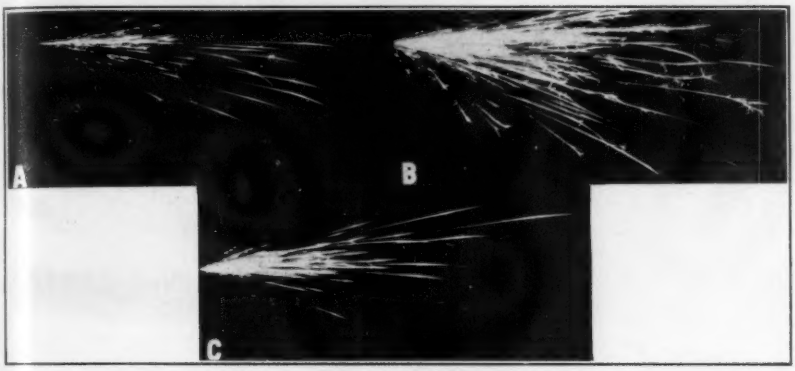

FIGURE 20.—*Typical features of spark streams of S.A.E. molybdenum steels.*

A, S.A.E. 4130 steel, spearpoints; B, S.A.E. 4150 steel, spearpoints, compact burst; C, S.A.E. 4615 steel, spearpoints, nickel spark, suppressed carbon burst.

FIGURE 21.—*Spark stream typical of molybdenum steel, showing spearpoints. Type S.A.E. 4130, c.f. (fig. 4).*

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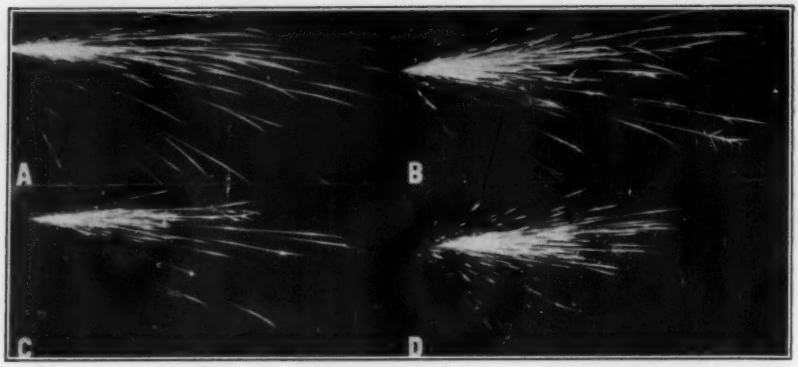

FIGURE 22.—*Typical features of spark streams of special molybdenum steels.*

A, 0.15 C Mn-Mo steel, suppressed carbon burst, spearpoints; B, steel M1 (0.20 C, 3.64 Ni, 0.07 Mo), spearpoint; C, steel M11 (0.53 C, 1.02 Si, 0.40 Mo), spearpoint, suppressed carbon burst; D, steel M16 (0.71 C, 5.75 Mo), spearpoints, suppressed carbon burst.

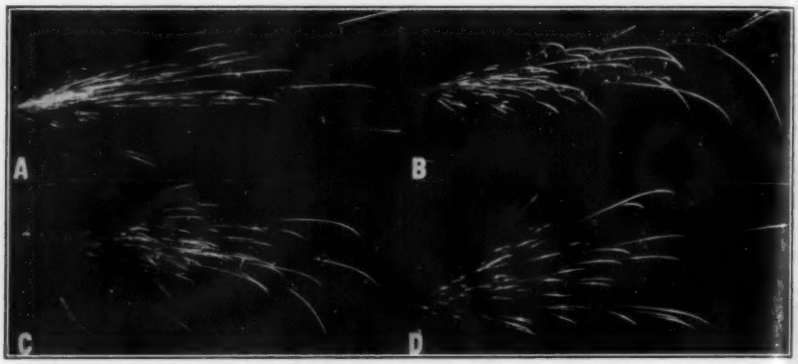

FIGURE 23.—*Typical features of spark streams of low-tungsten steels.*

A, Steel W1 (0.85 C, 0.45 W), heavy tips, suppressed carbon burst, dark carriers; B, steel W5 (1.25 C, 1.00 W), downward curve of tongues, heavy-tip wild bursts, suppressed carbon burst, red color; C, steel W10 (1.20 C, 1.60 W), downward curve of tongues, heavy tips, dark-red color, secondary carriers very dark, carbon burst suppressed; D, steel W12 (0.50 C, 2.00 W), carriers very dark, tongues bright, downward curve to tongues, suppressed carbon burst, very dark secondary carriers, bright buds and stars.

FIGURE 24.—*Typical features of spark streams of high-tungsten steels.*

A, Steel W22 (1.54 C, 1.00 Cr, 4.98 W), dark-red carriers, suppressed bursts, very dark secondary carriers, heavy tips, curved downward; B, steel W27 (0.68 C, 4.47 Cr, 2.20 V, 14.57 W), dark-red disjointed lines with an occasional swelling forming a tongue; C, steel W29 (0.73 C, 4.50 Cr, 1.22 V, 17.50 W), dark-red disjointed lines with an occasional carbon burst; D, same as C showing a very good example of a wild burst characteristic of tungsten.

TABLE 6.—*Composition of molybdenum steels studied*

Designation	C	Mn	P	S	Si	Ni	Cr	Mo	V	Cu	Fig. no.
	Per-cent	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	
S.A.E. 4130	0.29	0.62	0.019	0.031	0.21	------	0.69	0.22	------	------	20, 21
S.A.E. 4150	.47	.77	.022	.022	.23	------	.97	.19	------	------	20
S.A.E. 4615	.15	.62	.017	.024	.20	1.82	.30	.23	------	------	20
M 1	.20	.50	.011	.024	.10	3.64	.11	.07	------	------	22
M 2	.78	1.74	.016	.010	.47	.06	------	.08	0.22	------	------
M 3	.14	.94	------	------	.66	19.75	25.48	.16	------	------	------
M 9	1.00	.25	------	------	.25	------	1.00	.35	------	------	------
M 11	.53	.41	.018	.015	1.02	.14	.13	.40	------	------	22
M 13	1.42	.25	.013	.034	.50	------	4.20	.78	------	0.37	------
M 14	1.50	------	------	------	------	12.00	------	1.00	------	------	------
M 15	.06	.47	.016	.018	.42	9.84	19.65	3.07	------	.11	------
M 16	.71	.28	------	------	.28	------	------	5.75	------	------	22

All of the molybdenum steels studied showed a peculiar flare at the end of each ray. This was completely detached from the ray tip and is referred to here as the "spearpoint" (figs. 20 and 22). The color of the spearpoint varies with the steel. In the S.A.E. steels the spearpoint appeared in all cases in which the carbon content did not exceed 0.50 percent. The identification of such steels is simple and sure.

In the chromium-molybdenum steels the flower-like burst characteristic of chromium was evident. In other cases the carbon content appeared to be the factor which determined the predominating characteristic of the spark.

Of the 16 special molybdenum steels examined, all but 6 (M 2, M 3, M 9, M 13, M 14, M 15) showed the characteristic spearpoints in the spark stream. The failure of these six to show this feature can be attributed either to excess chromium, nickel, or carbon. The influence of carbon on the spearpoint is dependent on the molybdenum content; for example, in the steel (M 2) containing 0.78 percent carbon and 0.08 percent molybdenum, no spearpoints were observed in the spark stream, whereas steel M 16 (0.71 percent carbon and 5.75 percent molybdenum), showed spearpoints in the spark stream as did also steel M 1, containing 0.20 percent carbon and 0.07 percent molybdenum.

TABLE 7.—*Composition of tungsten steels studied*

Designation	C	Mn	Si	Cr	Ni	V	W	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
W 1	0.85	------	0.25	1.55	------	------	0.45	23
W 5	1.25	------	------	.20	------	------	1.00	23
W 10	1.20	------	------	.40	------	0.10	1.60	23
W 12	.50	------	------	1.65	------	.25	2.00	23
W 22	1.54	0.39	.25	1.00	------	------	4.98	24
W 27	.68	.24	------	4.17	0.28	2.20	14.57	24
W 29	.73	.27	.23	4.15	.02	1.22	17.50	24

7. TUNGSTEN STEELS

Table 7 lists the compositions of the tungsten steels which produced the representative spark streams illustrated in figures 23 and 24. Thirty additional steels containing tungsten were also studied, some

containing 0.5 to 2.5 percent tungsten, and manganese up to 1.5 percent; some with 1 to 2 percent tungsten and 2 percent carbon and others with 1 to 4 percent tungsten and 10 to 20 percent chromium. A special tungsten alloy examined contained 62 percent nickel, 12.5 percent chromium, 2.57 percent tungsten, 1.5 percent manganese, and 0.29 percent carbon.

Tungsten was found to impart a very characteristic red color to the spark stream in which the main carbon bursts were suppressed and the secondary carriers which were dark and inconspicuous were tipped with buds and stars the brilliance of which depended on the carbon content. The short, stubby tongues having a pronounced downward curvature were also characteristic. The spark streams of the steels having a low alloy content were full and dark and showed the characteristics just described. In steels containing 1.50 percent tungsten or more, the intensity of the carbon burst was diminished and the stream was dark red. In the steels of 2 percent tungsten content the carbon burst was variable even in the high carbon steels, in which the burst varied from one typical of a high carbon content to one associated ordinarily with a low carbon content. The presence of chromium in amounts of approximately 1.5 percent did not appear to affect the display, but larger amounts, for example, 10.5 percent, tended to "kill" the spark stream.

An increase in tungsten content above approximately 2 percent resulted in "wild" bursts in the spark stream which were sometimes so violent that the path of the pellet was entirely changed in direction. The carriers appeared as disjointed red lines.

The presence of 5-percent tungsten caused the spark shower to decrease rapidly in volume and steels containing 5 to 15 percent tungsten seldom showed more than dull red lines, the brilliance of which apparently depended largely on the chromium present. Steels containing 20 percent tungsten or more showed almost no spark stream. The combined chromium-tungsten content necessary for the elimination of the spark stream was much lower than that of nickel and chromium for producing a similar effect, the summation being approximately 20 percent.

8. SILICON-MANGANESE STEEL

Only one steel of the silico-manganese series was examined (S.A.E. No. 9250, containing 0.52 percent carbon, 0.83 percent manganese, 2.10 percent silicon). The spark stream (fig. 25) was dark red with club-shaped tongues and the carbon bursts were suppressed. This can be attributed largely to the influence of silicon. The manganese imparted a brilliant jacket to the burst.

9. MANGANESE STEELS

All the manganese steels considered (table 8) showed heavily "jacketed" bursts in the spark stream (fig. 26). The jacket was similar to that observed in spark streams of the nickel steels, although somewhat more brilliant. The carbon burst was apparently affected very little by the presence of manganese. The stream was full and even brighter than that of a corresponding plain carbon steel.

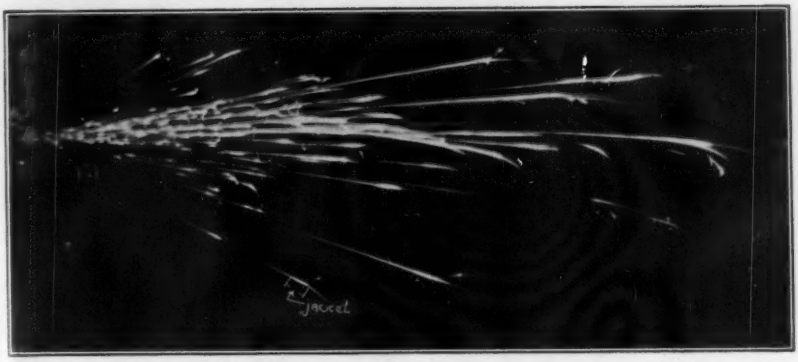

FIGURE 25.—*Spark stream typical of silico-manganese steel, showing jacketing effect. Type S.A.E. 9250.*

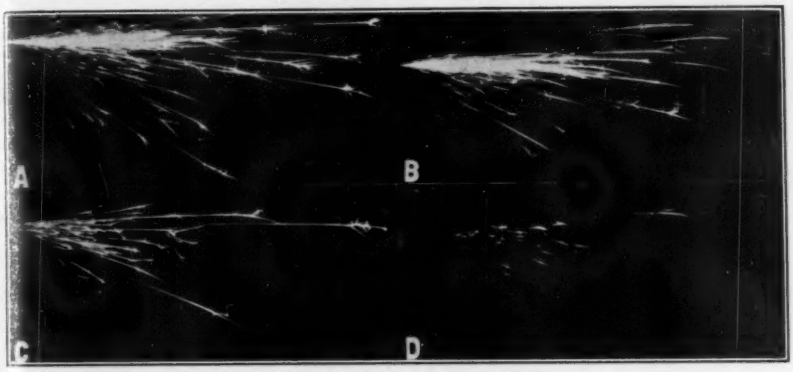

FIGURE 26.—*Typical features of spark streams of manganese steel and cast iron.*

A, Steel Mn1 (0.21 C, 1.00 Mn), jacket, compact burst; B, steel Mn5 (0.92 C, 1.60 Mn), jacket, compact burst, bright; C, steel Mn7 (1.00 C, 12.00 Mn), jacket, compact burst, bright; D, cast iron, very dark carrier, bright burst.

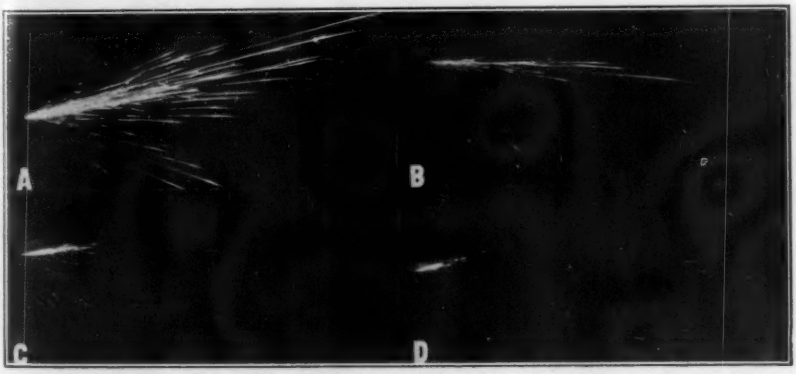

FIGURE 27.—*Spark streams showing the influence of nitriding time on type spark stream produced.*

A, Original steel, long full stream with spearpoints; B, same steel nitrided for 24 hours, stream shortened with a different burst; C, same steel nitrided for 48 hours, stream short, no burst; D, same steel nitrided for 72 hours, stream almost entirely suppressed, no burst.

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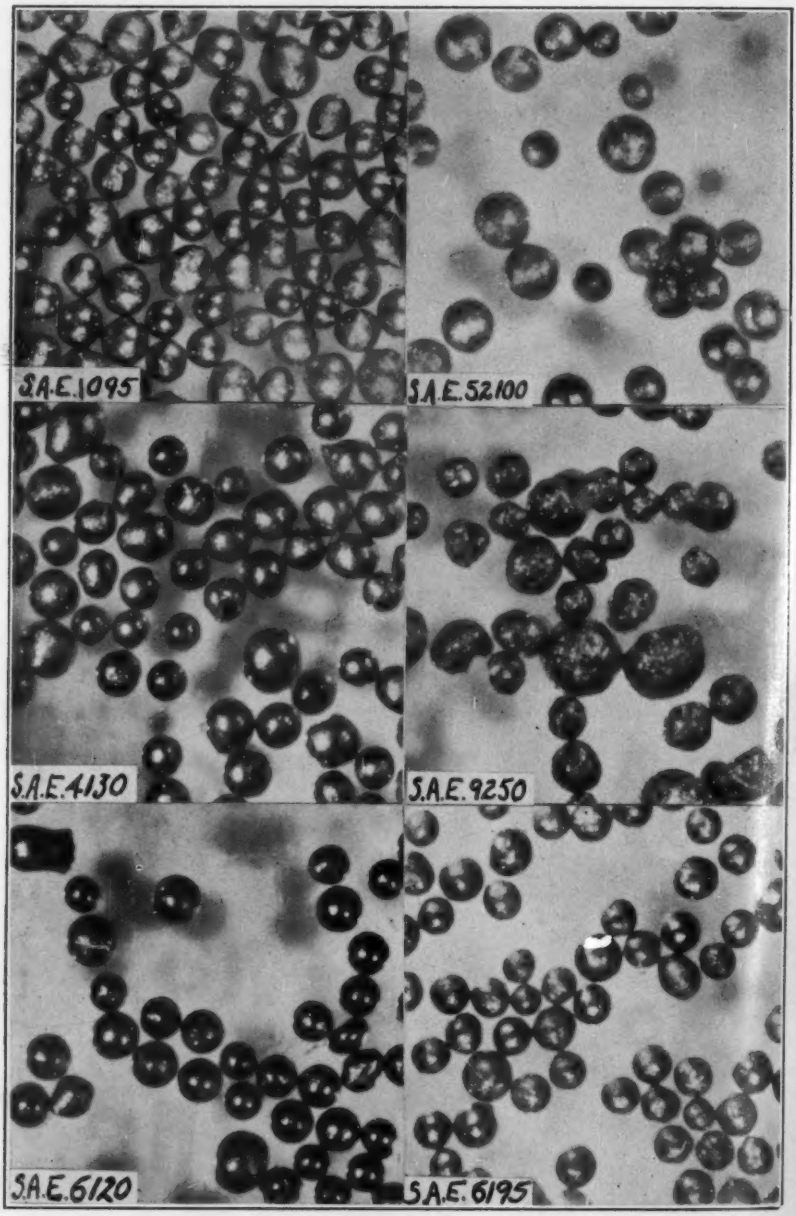


FIGURE 28.—*Typical pellet specimens* × *35.*

TABLE 8.—*Composition of manganese steels studied*

Designation	C	Mn	S	P	Si	Cr	Ni	Fig. no.
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
Mn 1	0.21	1.00	0.054	0.059	0.22	0.19	0.06	26
Mn 2	.39	.95	.090	.056	.21	.25	.10	------
Mn 3	.50	1.07	.033	.018	.21	.57	.14	------
Mn 4	.35	1.50	.040	.025	.45	------	------	26
Mn 5	.92	1.60	.012	.024	.21	.11	.12	------
Mn 6	.45	1.89	------	------	------	------	------	------
Mn 7	1.00	14.00	------	------	------	------	------	26

10. CAST IRON

Four plain cast irons were studied, with chief consideration given to their combined carbon contents (table 9). No marked differences were observed in the spark streams. In general, the stream consisted of very dark carriers with a comparatively light burst (fig. 26, *D*).

TABLE 9.—*Composition of cast irons studied*

Specimen number	Total carbon	Free carbon	Combined carbon	Mn	P	S	Si	Cr
				Percent	*Percent*	*Percent*	*Percent*	*Percent*
1-55	3.31	2.94	0.19	------	------	------	2.53	------
3-56	3.39	3.11	.28	------	------	------	2.51	------
4-56	3.45	3.08	.37	------	------	------	2.45	0.04
4-55	3.36	3.10	.26	0.72	0.515	0.039	2.30	.04

11. NITRIDED STEEL

A brief study of steels which has been nitrided, that is, "case hardened" by nascent nitrogen formed by the decomposition of ammonia, showed that the character of the spark stream was changed by nitriding and that this change was dependent upon the degree of nitriding. The photographs of figure 27 show the spark streams of the steel used (0.19 C, 0.38 Mn, 0.014 P, 0.012 S, 0.16 Si, 1.88 Al, 0.83 Mo) before nitriding and after 24, 48, and 72 hours nitriding, respectively. The possible usefulness of this method for determining differences in the thickness of the surface layer on nitrided steel products is apparent.

12. MISCELLANEOUS ALLOYS

A number of ferrous alloys were found to give no spark stream when tested in the ordinary manner. The compositions are shown in table 10.

TABLE 10.—*Composition of ferrous alloys showing no spark*

Manufacturer's designation	C	Mn	Si	Cr	Ni	Mo	V	W	Co
	Per-cent	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*	*Per-cent*
CN 2									
Dreadnaught	0.68	0.27	0.30	3.48			1.03	17.08	
G A	.55			15.00	35.00				
F	.55			15.00	25.00				
B 28	.66			26.00	8.00				
B	1.20			20.00	8.00				
C	.55			20.00	8.00				
B LC	.55			20.00	8.00				
N	.55			17.00	68.00				
CP 12									
V 3	.30			3.50			.50	10.00	
Cru EX 12	.75			3.50	.50		1.00	18.00	
A	.75			4.80		.80	.50	18.00	0.42
Cyclops B6	.68	.30		4.00				18.00	
WL 6	.67	.50	.126	1.49	.62		.17		
2825	.24	.35	.35	28.00					
55	.25	.70		28.96	.334				
44	.43	1.37		23.50	11.10				
V 2	.70			4.00			1.00	18.00	4.50
B 1	.70			4.00			1.00	18.00	4.50
Clarite									
Vulcan	.70	.40	.30	4.00			1.00	18.00	
Mercury	1.54	.39		1.00				5.00	
Rezistal 4	.20	.57	2.79	17.24	25.20				
Nichrome	.20	2.00		16.00	60.00				
Nichrome IV	.15		.25	19.50	77.50				
RE 3	.19	.58	1.34	24.50	20.00				

13. PELLET TEST

The possible identification of a steel by examination of the metal "pellets" which are formed during the spark testing of steel was first discussed by Hildorf and McCollam.[5] This test is supplementary to the spark test and not a complete one in itself. It has been found on examining the metallic "dust" from the spark stream of a steel that the individual burned particles are globules or pellets and that the pellets from one steel often differ quite characteristically from those of another kind of steel.

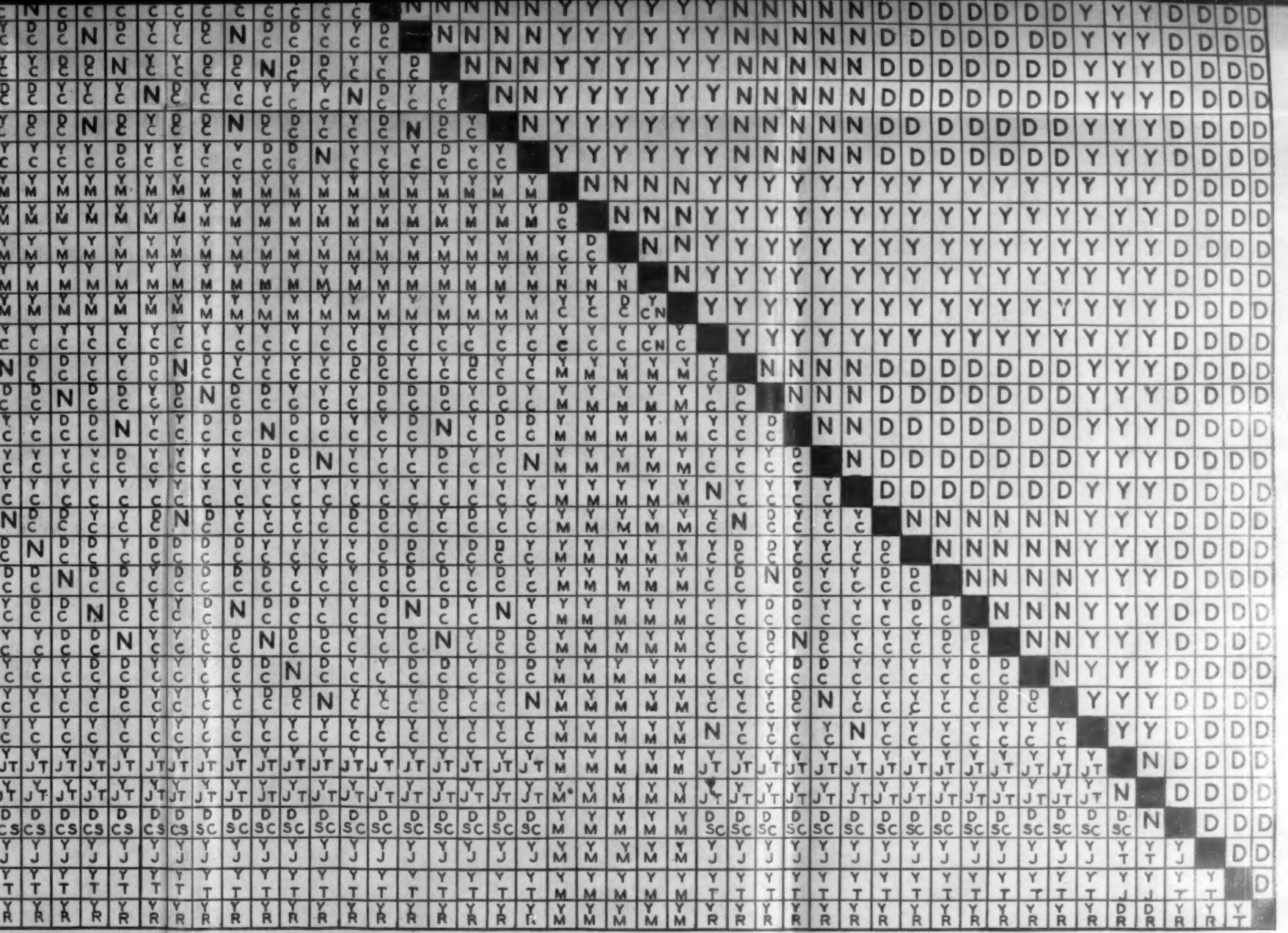
The "dust" from the spark streams in the foregoing examinations was collected and the spherical pellets separated from the more irregular particles by rolling on a sheet of paper. The spherical pellets were then sieved. All of the pellets which did not pass a 100-mesh screen were collected and examined under the microscope (x 35, fig. 28).

The pellets from plain carbon and nickel steels were shiny and black with indistinct patterns showing over the surface. The carbon content apparently did not affect the character of the surface of the pellet.

The pellets of chromium steels were rough and light gray in color. Those from molybdenum steel were very smooth and jet black. A scattering of elongated pear-shaped pellets was found among those collected from some vanadium steels. Silico-manganese steel produced badly blown pellets which, although spherical in shape, had holes blown entirely through them (table 11).

[5] Hildorf, W. G., and McCollam, C. H., Metal Pellets Produced by Spark Tests Used to Identify Alloy Steels, Iron Age, vol. 126, p. 1, 1930.

TABLE II.—Summary of point characteristics of S.A.E. steels



TEST

CT ON TONGUE

C- CARBON BURST

N-Ni SPARK

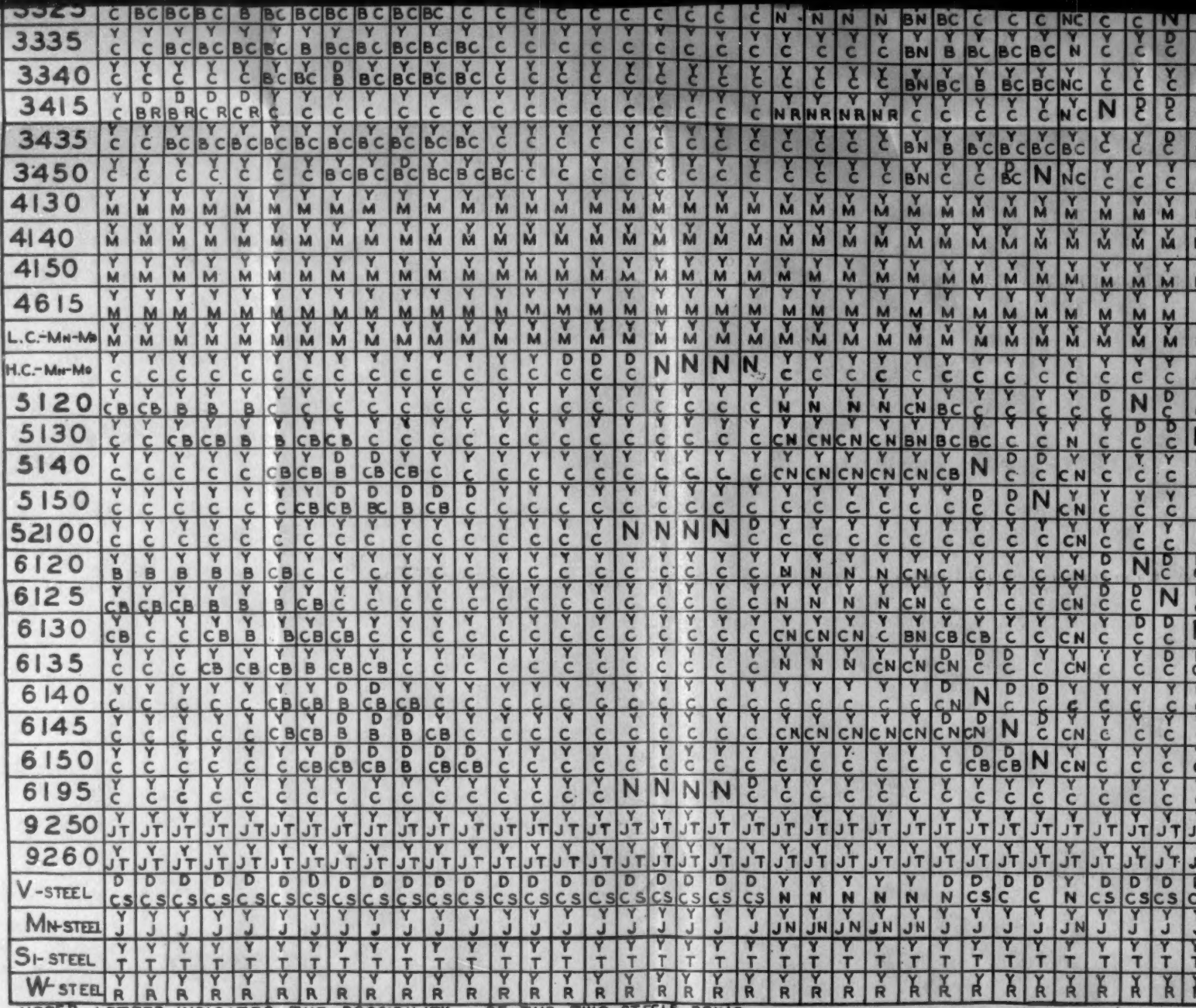
M- DETACHED SPEARPOINT

S- SPEARPOINT (NOT DETACHED)

ICIAL S.A.E. COMPOSITION.

R THE SPARK AND PELLET TESTS

29.—*Spark stream sorting characteristics*



UPPER LETTER INDICATES THE POSSIBILITY OF THE TWO STEELS BEING SORTED WHEN MIXED. LOWER LETTER INDICATES SPARK CHARACTERISTIC USED IN SORTING.

SPARK

Y- YES THE TWO STEELS INDICATED MAY BE SORTED.

N- NO THE TWO STEELS INDICATED CAN NOT BE SORTED.

D- MAY BE SORTED WITH DIFFICULTY.

B- CR BURST EFFECT

R- COLOR

T- Si EFFECT ON

J- JACKET.

A- NUMERICAL DESIGNATIONS ARE BASED ON THE S.A.E NUMBERING SYSTEM. NOT ALL STEELS SO DESIGNATED ARE OFFICIAL

SORTING CHARACTERISTICS OF STEEL FOR

8455—33. (Face p. 539.)

FIGURE 29.—*Sp*

PELLET TEST

SPARK TEST

	1085	1090	1095	10100	10130	2015	2115	2315	2320	2330	2335	2340	2345	2350	2512	3115	3120	3125	3130	3135	3140	3215	3220	3230	3235	3240	3245	3250	3312	3325	3335	3340	3415	3435	3450	4130	4140	4150	4615	L.C.-Mn-Mo	H.C.-Mn-Mo
1085																																									
1090																																									
1095																																									
10100																																									
10130																																									
2015																																									
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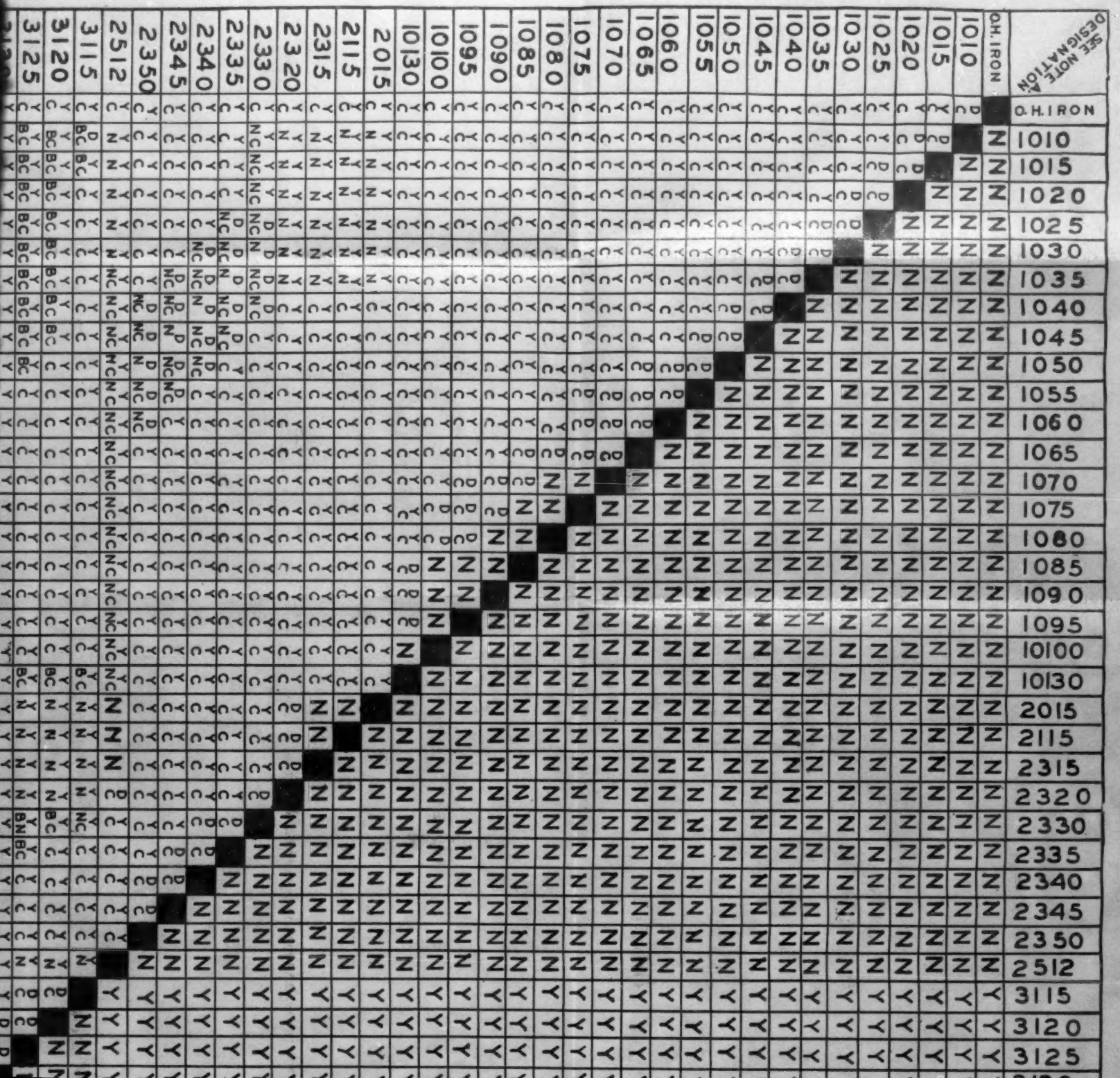
4130																																									
4140																																									
4150																																									
4615																																									
L.C.-Mn-Mo																																									
H.C.-Mn-Mo																																									

T TE S T

[illegible]	CD	CD	■	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3120
[illegible]	CD	■	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3125
[illegible]	■	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3130
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3135
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3140
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3215
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3220
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3230
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3235
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3240
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3245
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3250
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3312
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3325
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3335
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3340
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3415
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3435
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	3450
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	4130
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	4140
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	4150
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	4615
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	L.C-MN-Mo
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	H.C-MN-Mo
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	5120
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	5130
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	5140
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	5150
N	N	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	52100
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6120
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6125
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6130
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6135
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6140
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6145
D	D	D	D	D	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6150
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	6195
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	9250
Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	9260
D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	V-STEEL
D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	Mn-STEEL
D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	Si-STEEL
D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	D	W-STEEL

PELLET

TEST


O.H. IRON
SEE NOTE DESIGNATION
O.H. IRON
1010
1015
1020
1025
1030
1035
1040
1045
1050
1055
1060
1065
1070
1075
1080
1085
1090
1095
10100
10130
2015
2115
2315
2320
2330
2335
2340
2345
2350
2512
3115
3120
3125

PELLET

TEST

TABLE 11.—*Summary of pellet characteristics of S.A.E. steels*

Kind of steel	Pellet characteristics	
	Color	Shape and surface appearance
Plain carbon	Shiny black	Round and faintly patterned on a portion of the surface.
Nickel	do	Do.
Chromium	Light gray	Round and generally rough over entire surface.
Nickel-chromium	do	Do.
Chromium-vanadium	do	Round and generally rough over entire surface with a scattering of elongated pear-shaped pellets.
Molybdenum	Jet black	Round and very smooth having a tendency to be blown out on one side but still retaining round shape. An occasional elongated pear-shaped pellet appears.
Silicon-manganese	Dull black	Round but badly blown. Some holes entirely through the pellets.

IV. SUMMARY

1. SORTING CHART FOR S.A.E. STEELS

The data obtained in the study of the carbon and alloy steels, including S.A.E. steels, have been used as the basis of a sorting chart (fig. 29). In assembling this chart only the outstanding characteristics which can be depended upon as a basis of sorting groups of steels of known composition have been used. The character of the carbon burst has been used as much as possible. Cases where sorting is possible but may be difficult have been designated as "*D*". In all cases labeled "*Y*", there should be no difficulty in sorting the two steels if reasonable care is used. The alloy characteristics listed at the end of the chart apply only in a general way. They are the most probable characteristics, in addition to the carbon burst.

Half of this chart is devoted to the possible usefulness of the pellet test in the sorting of steels. It will be seen that in a number of cases the combination of the two tests will give conclusive evidence of the nature of the steel, whereas the indications of the spark test alone might be doubtful.

In general, it can be shown that out of possibly over 2,500 combinations of any two of the steels shown in this chart the identity of over 2,100 can be established. In addition, it may be possible to establish the identity in 390 additional combinations (listed under doubtful) and in the case of only 83 combinations are the results of the test entirely negative. The characteristics upon which this chart is based refer only to spark streams produced by the grinding wheel described and may not always apply if a distinctly different testing procedure is followed.

2. CONCLUSIONS

The spark test may be depended upon as a means for classifying steels into groups of similar composition, but cannot be successfully used as a means of identifying an unknown steel.

The spark test is probably the most rapid method for sorting mixed lots of steels containing two or three different compositions.

Definite spark stream characteristics were found in the steels studied for each of the alloying constituents; chromium, manganese, molybdenum, nickel, tungsten, and vanadium. The "carbon burst", however, is the most prominent feature of the spark stream.

The presence of chromium, molybdenum, silicon, or vanadium in some cases imparts characteristics to the fused particles or pellets resulting from the spark test which may be used in the identification of such steels.

V. ACKNOWLEDGMENTS

The author gratefully acknowledges to the steel companies the aid given in furnishing the various brands of special alloys; to Dr. V. Homerberg, of the Massachusetts Institute of Technology, in furnishing and nitriding the nitrided specimens, and to W. S. Rice, of the Bureau of Standards, for aid and advice in photographing the spark streams. Special acknowledgment is made of the cooperation and help given by C. H. McCollum, of the Timken Steel and Tube Co.

WASHINGTON, August 1, 1933.

U.S. DEPARTMENT OF COMMERCE, BUREAU OF STANDARDS

RESEARCH PAPER RP606

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

THE SYSTEM LIQUID IRON-CARBON OXIDES

By H. C. Vacher

ABSTRACT

The value 0.0025 previously obtained for the product of the carbon and oxygen contents of liquid iron under specified conditions has been confirmed. The method of approaching equilibrium consisted of passing a known mixture of carbon oxides at one atmosphere pressure over liquid iron maintained at 1,580° C. After approximately 90 minutes the liquid iron was allowed to freeze in the crucible and the ingot was analyzed for carbon and oxygen. Equilibrium was approached from both directions by adjusting the composition of the charges.

Additional experiments on the system liquid iron-hydrogen-water vapor showed that the data of both systems were in good agreement with data for the water-gas reaction.

CONTENTS

	Page
I. Introduction	541
II. Experimental Procedure	541
1. The System Liquid Iron-Carbon Oxides	543
2. The System Liquid Iron-Hydrogen-Water Vapor	546
3. Discussion	547
III. Summary	550

I. INTRODUCTION

In a previous paper [1] it was shown that the product of the concentrations of carbon and oxygen, for a number of ingots representing liquid iron at 1,620° C. and in contact with carbon oxides, was a constant within the limits of experimental error for a single ingot. The average value of the carbon-oxygen products obtained was 0.0025, which is approximately twenty times larger than the only other direct experimental value.[2] However, the value 0.0025 agrees with values [3] obtained from samples of basic open-hearth steels taken during the process of refining when the rate of carbon elimination was small. This agreement was not sufficient to determine the accuracy of the value 0.0025, and experimental work has been continued. The work was extended to include a few experiments on the system liquid iron-hydrogen-water vapor in order that the accuracy of the data obtained could be judged by correlation with related equilibria.

II. EXPERIMENTAL PROCEDURE

The method of attaining equilibrium consisted of passing a known mixture of carbon oxides, at one atmosphere pressure, over liquid iron maintained at a temperature of 1,580° C. After approximately 90

[1] H. C. Vacher and E. H. Hamilton, Carbon-oxygen equilibrium in liquid iron. Trans. Am. Inst. of Mining & Metallurgical Engrs., vol. 95, pp. 124–140, 1931.

[2] A. B. Kinzel and J. J. Egan, Experimental data on the equilibrium of the system iron-oxide-carbon in molten iron. Trans. A.I.M.M.E., Iron and Steel Division, pp. 300-319, 1929.

[3] C. H. Herty, Jr., Discussion. Iron and Steel Technology, A.I.M.M.E., p. 131, 1928.

minutes' exposure to the gases, the liquid iron was allowed to solidify in the crucible and the resulting ingot was analyzed for carbon and oxygen.

The furance arrangement was essentially the same as used in the previous work.[4] A magnesia crucible containing the iron was placed in a round-bottom alundum thimble which rested on a magnesia block containing a rounded depression. This facilitated centering the crucible and bringing the top surface of the charge in line with the guide tube and the eyepiece of the furnace top. The temperature was measured by means of an optical pyrometer of the disappearing filament type. The pyrometer was calibrated under black-body conditions to ±10 degrees. As the temperature readings during an experimental run were made on the surface of the liquid iron it was necessary to correct the temperature corresponding to the calibration for emissivity. Black-body conditions necessary for the determination of this correction were obtained by means of a crucible that contained a 3 millimeter bore tube centered to coincide with the lognitudinal axis of the crucible. The length of the tube which was heated by the charge was 22 or more millimeters. In order that the bottom of the hole would be at the temperature of the charge the tube was filled with magnesia sand to a depth of 7 millimeters above the bottom of the crucible. The emissivity correction was determined by taking alternate readings in the sight tube and on the surface of the liquid iron. With this furnace arrangement, pyrometer readings are reproducible within 2 milliamperes or 15 degrees.

Each mixture of carbon monoxide and carbon dioxide used was prepared by passing carbon dioxide, from a cylinder, through crushed graphite which was held at a predetermined temperature. The amount of carbon dioxide in the mixture of carbon oxides was controlled by varying the temperature of the graphite, for example, if the graphite was maintained at 1,050° C., the resultant gas mixture contained about 11 percent carbon dioxide. Lower temperatures increased and higher temperatures decreased the carbon dioxide content. For a given temperature, the composition of the mixture could be varied to some extent by modifying the rate of flow of gas through the hot graphite. Analyses of several samples showed that the prepared mixtures contained not less than 99 percent carbon oxides, with about 0.3 percent nitrogen and 0.7 percent hydrogen as impurities. The gas mixture was stored over a saturated brine solution and the hydrostatic pressure of the displaced solution was employed to force the gas through drying towers and through the furnace in a subsequent experimental run. Hydrostatic pressure equivalent to 2 or 3 cm of mercury was sufficient to maintain a steady flow of gas through the furnace.

It was found that the carbon dioxide content of a reservoir full of mixed gases decreased steadily as the reservoir was emptied, presumably on account of the selective solubility of carbon dioxide in the brine solution. For example, an initial carbon dioxide content of 12 percent might drop to 11.5 percent by the time the reservoir was nearly empty. This progressive decrease in the carbon dioxide content necessitated sampling of the gas stream during a run to determine the average composition of the gas entering the furnace. In these control analyses only the amount of carbon dioxide in the sample was determined.

[4] See footnote 1.

Examination of the furnace after several runs showed that no soot had deposited in the silica tube used to pass the entering gases over the surface of the iron. Consequently, the composition of the gas mixture which came in contact with the liquid iron was the same as that of the gas mixture as it entered the furnace. However, the deposition of small amounts of soot on the cold parts of the furnace near the gas exit indicated that some decomposition of the carbon monoxide was taking place after it was in contact with the liquid iron. This phenomenon, and the fact that the change in composition of the gas phase, due to the reaction with liquid iron, at best was too small to be readily detected, made it useless to attempt to follow the course of the gas-liquid iron reaction by comparing the composition of the exit gases with that of the entering gases.

In the absence of a definite test to determine whether equilibrium had been reached for any particular ingot, two or more ingots were prepared with each mixture of carbon oxides. One of the ingots was prepared from a charge of high initial oxygen content, the other from a charge of low oxygen content. Equilibrium therefore was approached from both the high-oxygen and low-oxygen sides. Also, in some cases additional ingots were prepared to represent approach to equilibrium from both the high-carbon and low-carbon sides. If equilibrium were attained in the time allotted, 1½ to 2 hours, the carbon (or oxygen) contents of a pair of ingots should agree. However, if the carbon (or oxygen) contents of a pair of ingots do not agree, the equilibrium value should lie within the limits of the pair of experimental values. This method of determining the carbon (or oxygen) content at equilibrium with a known mixture of carbon oxides obviously is accurate only in the case where the carbon (or oxygen) contents of the pair of ingots agree. However, in case an agreement is not obtained, a reliable value for the equilibrium content can be had by averaging the two carbon (or oxygen) contents, provided the difference between the pair of ingots is small.

The base material for the charges consisted of pieces of open-hearth iron cut from a one-inch round rod. The iron contained 0.07 percent oxygen, 0.003 percent alumina and 0.002 percent silica, and each piece weighed approximately 100 grams.

1. THE SYSTEM LIQUID IRON-CARBON OXIDES

In the first series of runs, made with gas mixtures which contained less than 10 percent carbon dioxide, gases were evolved from the metal during solidification. The evolution of gas increased in violence as the carbon-dioxide content of the mixture decreased from about 10 percent to 5 percent or less. Figure 1 shows an ingot prepared under a gas mixture containing 5 percent carbon dioxide. It was necessary to add aluminum to all charges prepared under gas mixtures containing less than 10 percent carbon dioxide in order to retain, as aluminum oxide in the solid metal, the oxygen present in the liquid metal. The amount of this aluminum oxide was determined by means of the hydrochloric-acid residue method, but segregation of the aluminum oxide complicated the problems of analysis. The results for ingots 2 and 3 in table 1 illustrate the failure to obtain duplicate results in the analyses of duplicate ingots. The average values for the carbon-oxygen products, derived from table 1, are 0.0021 for gas mixtures containing 1.5 percent carbon dioxide and

0.0028 for 7 percent carbon dioxide. However, all of the data in table 1, including these computed carbon-oxygen products, are open to question on the grounds of the analytical difficulties which resulted from segregation of alumina.

TABLE 1.—*Carbon and oxygen contents of ingots prepared in contact with carbon oxides containing less than 10 percent carbon dioxide*

Ingot no.	CO_2 in gas mixture	Charge	Carbon	Oxygen	Carbon-oxygen product
	Percent		*Percent*	*Percent*	*Percent*
1	1.4	Open-hearth iron melted in a vacuum	0.09	0.016	0.0014
2	1.5	Open-hearth iron plus 0.3 percent graphite	.17	.019	.0032
3	1.7	do	.19	.014	.0027
4	6.9	Open-hearth iron plus 0.2 percent graphite	.05	.051	.0025
5	7.0	Open-hearth iron melted in a vacuum	.04	.077	.0031

A second series of ingots was prepared under mixtures of carbon oxides containing more than 10 percent carbon dioxide. These ingots did not evolve gas during solidification. Therefore, a determination of the carbon and oxygen contents of these ingots represents the carbon and oxygen contents of the liquid iron. Preliminary experiments with gas mixtures containing about 12 percent carbon dioxide showed that the oxygen contents of the ingots would be approximately 0.15 percent. It was necessary, therefore, to prepare charges containing more than 0.15 percent oxygen in order that the final oxygen content might be approached by a reduction in the oxygen content of the charge. These charges of high oxygen content were prepared by exposing liquid iron to a stream of carbon dioxide, ingots containing from 0.2 to 0.5 percent oxygen being obtained.

It was observed that when an ingot which contained more than 0.1 percent oxygen was melted in a magnesia crucible, the surface of the melt remained bright and clean but there was appreciable formation of slag on the surfaces of the crucible in contact with the metal, as shown in figure 2. This indicated a reaction of magnesia with iron oxide from the liquid iron. This reaction tends to decrease the oxygen content of the melt and, if it continued throughout the run, equilibrium between the liquid iron and carbon oxides might not be attained. However, the continued formation of slag throughout the run is improbable. In the high-frequency furnace, there is a sharp temperature gradient between the liquid metal and its refractory container, which results in solidification of the outer surface of the slag layer, thereby preventing further slag formation. This leads to the belief that the formation of this slag does not affect the final equilibrium content of oxygen although it does affect the rate at which equilibrium is attained.

This belief is confirmed by experiments in which beryllia crucibles were used in place of magnesia. Beryllia did not form a slag with FeO under the conditions of these experiments as is illustrated by the photograph of an ingot melted in a beryllia crucible (fig. 3). Ingots 10 and 11 in table 2 show the results of duplicate runs, in magnesia and beryllia crucibles. It will be shown subsequently that equilibrium probably was not attained in any of the runs in the time allowed. However, in ingot 10 the effect of the formation of the slag would be to

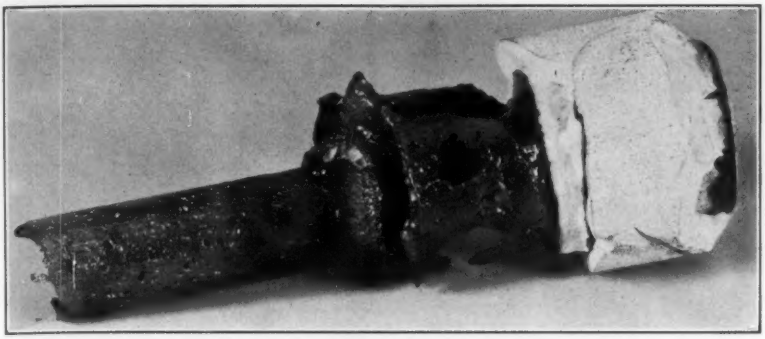


FIGURE 1.—*Result of evolution of gas during solidification of molten iron prepared under a mixture of carbon oxides containing 5 percent carbon dioxide.*

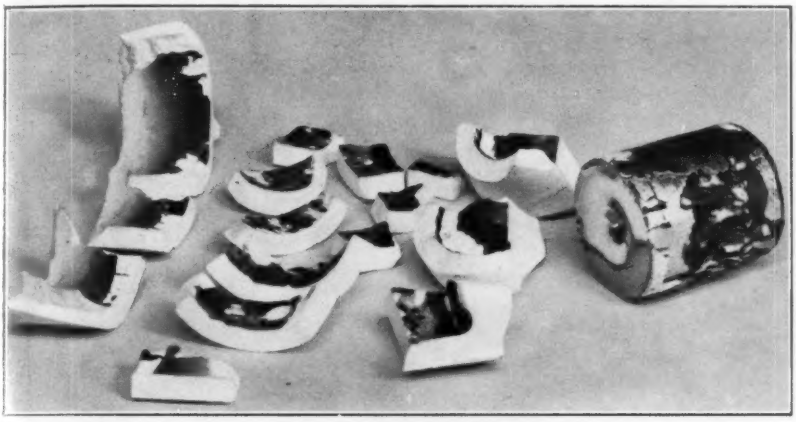


FIGURE 2.—*Ingot prepared in a magnesia crucible.*

FIGURE 3.—*Ingot prepared in a beryllia crucible.*

increase the rate of approach to equilibrium from the high oxygen side; hence, the oxygen content of ingot 10 should be lower, and nearer to the equilibrium value, than in the case of ingot 11, where the oxygen content was lowered only through the action with the gas mixture. Although the two are in reasonable agreement, the oxygen content of ingot 10 actually is lower than that of ingot 11. This is in accord with the assumption that the formation of a MgO-FeO slag affects the rate of approach to equilibrium but does not affect the final equilibrium conditions. The oxygen content of ingots 6, 7, 8, and 9 cannot be compared in regard to the formation of a MgO-FeO slag, because each was prepared under mixtures of carbon oxides of different composition.

TABLE 2.—*Carbon and oxygen contents of ingots prepared in contact with mixtures of carbon oxides containing more than 10 percent carbon dioxide*

[NOTE.—The letters T, C, and B refer to the top, center, and bottom portions of the ingots]

Ingot no.	CO_2 in gas mixture	Charge	Refractory	Sample	Carbon	Oxygen	Carbon-oxygen product
	Percent				*Percent*	*Percent*	
6	10.3	Open-hearth iron	Magnesia	T	0.016	0.107	0.0021
				C	.018	.104	
				B	.026	.111	
7	12.1	do	Beryllia	T	.018	.127	.0021
				C	.014	.131	
				B	.019	.117	
8	14.3	Open-hearth iron fused in a vacuum	Magnesia	B	----------	.168	----------
9	16.7	do	do	B	----------	.174	----------
10	11.3	Oxidized open-hearth iron	do	T	.021	.139	.0026
				C	.018	.137	
				B	.018	.138	
11	11.2	do	Beryllia	T	.018	.156	.0029
				C	.017	.151	
				B	.022	.158	

The results for the series of ingots prepared under gas mixtures which contained more than 10 percent carbon dioxide are shown in table 2. In order to obtain reliable values for the carbon and oxygen contents of ingots of this series, each ingot was cut laterally into three discs. Half of each disc was used for a carbon determination and the remaining half for an oxygen determination by the vacuum fusion method. In computing the carbon-oxygen product for an ingot the averages of the three carbon and of the three oxygen determinations were used. Ingots 6, 7, 10, and 11 were prepared under approximately the same gas compositions. Ingots 6 and 7 were prepared from charges containing a low initial oxygen content, whereas ingots 10 and 11 were obtained from charges with high initial oxygen contents. Ingot 10 is believed to approach equilibrium more nearly than does ingot 11, for reasons already discussed. The carbon-oxygen product obtained for molten iron under mixtures of carbon oxides containing approximately 11 percent carbon dioxide therefore is 0.0023. This value is obtained by taking the numerical average of the products for ingots 6 or 7 and 10. The value 0.0023 is in good agreement with the value 0.0025 obtained in previous work.[5] It is also in qualitative agreement with the results shown in table 1. It is evident that a value of the order of magnitude of 0.0025, for the carbon-oxygen product in liquid iron at about 1,600° C. and one atmosphere pressure, is a reproducible value. The accuracy of this

[5] See footnote 1.

value will be demonstrated in the subsequen t discussion of the results obtained for the system liquid iron-hydrogen-water vapor.

2. THE SYSTEM LIQUID IRON-HYDROGEN-WATER VAPOR

The same general procedure that was employed in the experiments with carbon oxides was used in preparing a few ingots under hydrogen which contained approximately 4 percent water vapor. The hydrogen-water vapor mixture was made by passing a stream of purified hydrogen through a saturated sodium chloride solution. The saturated brine solution was used in order that the partial pressure of water vapor in the hydrogen-water vapor mixture would be less than the vapor pressure of water at room temperature. This prevented water from forming in the tubing that led the gases to the furnace. The hydrogen-water vapor mixture was passed through the furnace at a rate of 150 ml per minute for a period of from 1½ to 2 hours.

In two runs the original charge consisted of pieces of open hearth iron which contained 0.07 percent oxygen. In these two runs therefore, equilibrium in the metal was approached from the high oxygen side. In two additional runs equilibrium was approached from the low oxygen side through the use of charges of open hearth iron which had been held molten under a stream of hydrogen for one half hour. Previous experiments had shown that this treatment reduced the oxygen content to about 0.005 percent. At the start of each experimental run the charge was melted in vacuo. This resulted in almost complete elimination of the small amount of carbon present in the ingot iron, accompanied by a slight reduction in oxygen. It was assumed that the carbon content of the charges after this treatment was too low to exert any effect on the iron-hydrogen-oxygen equilibrium. These charges after the 1½ to 2-hour exposure to the hydrogen-water vapor mixture at 1,580° C. did not evolve gas during solidification. However, several attempts to prepare ingots at 1,620° C. did result in the evolution of a small amount of gas during solidification.

TABLE 3.—*Oxygen and hydrogen contents of ingots prepared in contact with mixtures of hydrogen and water*

[NOTE.—The letters T and B refer to the top and bottom portions of the ingots]

Ingot no.	Water vapor in gas mixture	Charge	Sample	Oxygen	Hydrogen
	Percent			*Percent*	*Percent*
12	3.9	Open hearth iron fused and kept in contact with a stream of H_2 for ½ hour	T	0.011	0.0003
			B	.008	.0003
13	4.2	Open hearth iron	T	.013	.0001
			B	.009	.0003
14	4.3	Same as no. 12	T	.011	.0001
			B	.012	.0004
15	3.4	Same as no. 13	T	.011	.0001
			B	.009	.0001

The results of the analyses of the ingots prepared under hydrogen-water vapor at 1,580° C. are shown in table 3. Top and bottom samples of each ingot were analysed for oxygen. The results show the absence of serious segregation.

3. DISCUSSION

In previous work on the system liquid iron-carbon oxides the data obtained have been open to question for two reasons: First, uncertainty on account of experimental difficulties; second, uncertainty whether or not the data represented equilibrium. The first uncertainty has to do with the reproducibility of the data under the conditions of the experiment, the second with the accuracy of the data, at least in regard to the order of magnitude.

The present data have yielded an average carbon-oxygen product of 0.0023 for liquid iron at about 1,600° C. and under one atmosphere of carbon oxides. A value of 0.0025 was obtained under similar conditions in previous work. These values were obtained from ingots which were prepared from charges of high and low oxygen contents and from high and low carbon contents and under mixtures of carbon oxides of various compositions. Therefore, it is concluded that the values, 0.0023 and 0.0025, can be reproduced.

The accuracy of any data representing an equilibrium is established when the data can be shown to be in agreement with other equilibria. In the following discussion it will be shown that the data in tables 2 and 3 are in good agreement with accepted data for the well-known water-gas reaction and that the data in table 2 are in qualitative agreement with accepted data on the Fe−C system.

The first equilibrium constant to be derived is the constant for the reduction of iron oxide by carbon monoxide according to equation (1).

$$\text{FeO (in solution)} + \text{CO (gas)} = \text{Fe (liq.)} + \text{CO}_2 \text{ (gas)};$$

$$K_1 = \frac{N_{Fe}}{N_{FeO}} \cdot \frac{p_{CO_2}}{p_{CO}} \qquad (1)$$

The computation of this constant and subsequent constants from the data in table 2 necessitates the assumption that the free iron in the liquid iron solution at these carbon contents is essentially equal to 1 minus N_{FeO} minus N_{Fe_3C}, where N is the mol fraction. The assumption is based on the following considerations: (a) the presence of appreciable amounts of carbon monoxide and carbon dioxide in liquid iron is improbable;[6] (b) the oxygen content of liquid iron can be considered as equivalent to ferrous oxide, since it has been shown that the ratio of iron oxide in a lime slag to the oxygen content of liquid iron is a constant;[7] (c) the carbon in liquid iron is present as iron carbide that dissociates slightly at high carbon contents according to the reaction $Fe_3C = C + 3Fe$.[8]

The data for the computation of K_1 are shown in table 4. In computing the mol fractions the average values for the carbon and oxygen contents of each ingot listed in table 2 have been used. The first four values for K_1 are computed from the results of runs in which equilibrium was approached from the low oxygen side. If equilibrium were not attained, the final oxygen content would be lower

[6] V. H. Gottschalk and R. S. Dean, The Solubility of Gases in Metals. A.I.M.M.E. Preprint, February 1932, 16 pages.

[7] C. H. Herty, Jr., et al., The Physical Chemistry of Steel-making. The Solubility of Iron Oxide in Iron. Min. & Met. Invest. Bull. 34, U.S. Bureau of Mines, Carnegie Inst. of Tech. and Mining & Met. Advisory Boards (1927).

[8] Friedrick Körber and Willy Oelsen, Thermodynamic Considerations of Several Equilibrium Curves of the System Iron-Carbon. (Thermodynamisches Betrachtungen Zu einigen Gleichgewichtskurven des Zustandsschaubildes Eisen-Kohlenstoff). Archiv. für das Eisenhüttenwesen, vol, 5. pp. 569-578, May 1932. Extended abstract by J. B. Austin, Metals and Alloys, vol. 4, pp. 49-53; April 1933.

than the equilibrium concentration and the value for the constant K_1 would be higher than the correct value. On the other hand, in runs 5 and 6 equilibrium was approached from the high oxygen side. If equilibrium were not attained in these two runs, the final oxygen content would be higher than the equilibrium concentration and the value for K_1 would be correspondingly low. The calculated values for K_1 in table 4 indicate that equilibrium was not fully attained in the time allowed. Consequently, the true value for K_1 is considered to lie between the lowest value obtained in the first four runs, 28.2, and the highest value obtained in the last two runs, 26.2. The numerical average of these two values, 27.0, was used in subsequent computations as the value of K_1 at 1580° C.

TABLE 4.—*Computation of equilibrium constants K_1 and K_4*

Ingot no.	N_{Fe_3C}	N_{FeO}	N_{Fe}	p_{CO_2} atm.	p_{CO} atm.	N_{Fe}/N_{FeO}	p_{CO_2}/p_{CO}	K_1	N^3_{Fe}/N_{Fe_3C}	p^2_{CO}/p_{CO_2}	K_4
6	0.00094	0.00374	0.995	0.103	0.897	266	0.115	30.6	1052	7.81	8220
7	.00079	.00444	.995	.121	.879	224	.138	29.9	1247	6.39	7970
8	-------	.00588	.993	.143	.857	169	.167	28.2	-------	-------	-------
9	-------	.00609	.993	.167	.833	163	.200	32.6	-------	-------	-------
10	.00089	.00483	.994	.113	.887	206	.127	26.2	1110	6.96	7730
11	.00089	.00543	.994	.112	.888	183	.126	23.1	1110	7.04	7810
K_1 average of 8 and 10								27.0	K_4 average		7930

The constant K_1 can be combined with the data for the liquid iron-hydrogen-water vapor system, listed in table 3, to yield a constant for the familiar water-gas reaction. Equilibrium in the iron-hydrogen-water vapor system is represented by equation (2)

$$FeO\ (\text{in solution}) + H_2\ (\text{gas}) \rightleftarrows Fe\ (\text{liq.}) + H_2O\ (\text{gas});\ K_2 = \frac{N_{Fe}}{N_{FeO}} \cdot \frac{p_{H_2O}}{p_{H_2}} \quad (2)$$

and K_2 divided by K_1 is equal to K_3, the constant for the water gas reaction (3).

$$CO_2\ (\text{gas}) + H_2\ (\text{gas}) \rightleftarrows CO\ (\text{gas}) + H_2O\ (\text{gas});\ K_3 = \frac{p_{CO}}{p_{CO_2}} \cdot \frac{p_{H_2O}}{p_{H_2}} = \frac{K_2}{K_1} \quad (3)$$

TABLE 5.—*Computation of equilibrium constant K_2.*

Ingot no.	N_{FeO}	N_{Fe}	p_{H_2O} atm.	p_{H_2} atm.	N_{Fe}/N_{FeO}	p_{H_2O}/p_{H_2}	K_2
12	0.000350	0.999	0.039	0.961	2854	0.0406	116
13	.000381	.999	.042	.958	2622	.0438	115
14	.000420	.999	.043	.957	2379	.0449	107
15	.000350	.999	.034	.966	2854	.0352	101
K_2—Average							110

The computed values for K_2, in table 5, average 110 for four ingots; the value for K_1 in table 4 is 27.0; the value derived for K_3, therefore, is 4.07. The value for the water-gas constant at 1,580° C. is 3.95 computed with the aid of Chipman's free energy equation.[9] The value for K_3 derived from the present work and from the free energy equa-

[9] J. Chipman, The Free Energy of Water, Carbon Monoxide, Carbon Dioxide, and Methane, Ind. & Eng. Chem., vol. 24, pp. 1013, 1932.

tion are considered to be in close enough agreement to demonstrate that the data in tables 2 and 3 agree with accepted data for the water-gas reaction.

The second constant to be derived from the data in table 2 is the constant for the oxidation of iron carbide by carbon dioxide according to equation (4).

$$Fe_3C\ (\text{in solution}) + CO_2(\text{gas}) \leftrightarrows 3Fe\ (\text{liq.}) + 2CO\ (\text{gas});\ K_4 = \frac{N^3_{Fe}}{N_{Fe_3C}} \cdot \frac{p^2_{CO}}{p_{CO_2}} \quad (4)$$

The average value obtained for K_4 is 7.9×10^3 according to the computations indicated in table 4.

If the relation between the carbon content of liquid iron and the partial pressures of carbon oxides as expressed by K_4 is true over the complete range of possible carbon contents, then the carbon content can be determined from a knowledge of the composition of a mixture of carbon oxides at one atmosphere pressure. The composition of carbon oxides at one atmosphere pressure in equilibrium with graphite can be obtained from the equilibrium constant of the producer-gas reaction (5).

$$C\ (\text{graphite}) + CO_2(\text{gas}) \leftrightarrows 2CO\ (\text{gas});\ K_5 = \frac{p^2_{CO}}{p_{CO_2}} \quad (5)$$

Inasmuch as the mixture of carbon oxides at one atmosphere pressure in equilibrium with graphite is also in equilibrium with liquid iron saturated with graphite, K_5 can be combined with K_4 to give the value of $\frac{N^3_{Fe}}{N_{Fe_3C}}$. The value of K_5 at 1,580° C. is 14.4×10^3 when computed with the aid of Chipman's free energy equation [10] for the producer-gas reaction. The value of N_{Fe}/N_{Fe_3C} when liquid iron is saturated with graphite therefore, is 0.55. The carbon content of liquid iron then can be computed from the value 0.55 by considering N_{Fe} equal to $1 - N_{Fe_3C}$. The value of N_{FeO} is negligible at high carbon contents. On this basis the carbon content corresponding to 0.55 is 4.4 percent. The carbon content of liquid iron saturated with graphite is 5.3 percent according to the iron-carbon diagram.[11] It is concluded therefore that K_4, is not applicable over the complete range of possible carbon contents. In order to correlate the data describing liquid iron saturated with graphite and the data in table 4 it must be assumed that iron carbide dissociates to some extent, according to equation (6).

$$Fe_3C\ (\text{in solution}) \rightleftharpoons 3\ Fe(\text{liq.}) + C\ (\text{in solution});\ K_6 = \frac{N^3_{Fe}}{N_{Fe_3C}} \cdot N_C \quad (6)$$

K_6 can be computed from the value of N^3_{Fe}/N_{Fe_3C}, 0.55 and the carbon content, 5.3, when liquid iron is saturated with graphite. This computed value for K_6, 0.038, can be used with K_1 and K_4 to derive the data for the dissociation of Fe_3C, at different carbon contents, recorded in table 6. It is evident that the dissociation of Fe_3C is negligible at very low carbon contents but becomes increasingly important as carbon contents of about 5 percent are reached.

[10] See footnote 9.
[11] See footnote 8.

TABLE 6.—*The carbon and oxygen contents of liquid iron in equilibrium with different mixtures of carbon oxides at one atmosphere pressure and 1,580° C computed from K_1, K_4, and K_6*

Carbon dioxide	Carbon (as Fe_3C)	Carbon (as C)	Total carbon	Oxygen	Carbon-oxygen product (C) x (O)	Phases present in addition to gas and liquid iron phases
Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	
21.1	0.008		0.008	0.28 [a]	0.0022	FeO (slag).
10.0	.022	0.00084	.023	.117	.0027	
6.0	.039	.0015	.041	.068	.0028	
2.0	.126	.0049	.131	.0216	.0028	
1.0	.249	.0098	.259	.0105	.0027	
0.5	.474	.0194	.493	.0049	.0024	
.1	1.656	.0856	1.742	.00067	.0012	
.05	2.434	.1553	2.589	.00029	.0007	
.007	4.4	.9	5.3 [b]	.000025	.0001	C (graphite).

[a] Oxygen content of liquid iron in equilibrium with iron oxide. See footnote 7.
[b] Carbon content of liquid iron in equilibrium with graphite. See footnote 8.

Körber and Oelsen[13] showed that the latent heat of fusion of pure iron can be correlated with the carbon content of the solidus and liquidus of the iron carbon diagram if it is assumed that the carbon content of the liquidus consists of Fe_3C slightly dissociated. In the preceding discussion it has been shown that the carbon contents of liquid iron and the corresponding composition of carbon oxides at one atmosphere pressure can be correlated if it is assumed that the carbon content of liquid iron consists of Fe_3C slightly dissociated. The two sets of data therefore lead to the same assumption and consequently can be concluded to be in qualitative agreement. A more quantitative comparison could be made if K_6 could be computed as a function of temperature from the iron-carbon diagram and the latent heat of fusion of pure iron, but the available data are not sufficiently reliable and the computations are too sensitive to errors in the data.

The constants, K_1, K_4, and K_6, have been used further to compute the variations in the carbon-oxygen product with varying composition of the carbon-oxides mixture at one atmosphere pressure for liquid iron at 1,580° C. These computations, recorded in table 6, show that the carbon-oxygen product is not a constant for all mixtures of carbon dioxide and carbon monoxide. Experimental work is practicable only with material which contains between 0.01 and 0.5 percent carbon and between 0.005 and 0.3 percent oxygen. It is evident that within these limits the carbon-oxygen product lies between 0.002 and 0.003. The average value of 0.0025 can be used throughout the range which can be investigated experimentally, since the error resulting from the use of this single value will be well within the limits of precision of the experimental procedure.

III. SUMMARY

The present data on the system liquid iron-carbon oxides have been used in computing the equilibrium constants of the following equations

$$FeO\ (\text{in solution}) + CO(\text{gas}) \rightleftarrows Fe(\text{liq.}) + CO_2(\text{gas});$$

$$K_1 = \frac{N_{Fe}}{N_{FeO}} \cdot \frac{p_{CO_2}}{p_{CO}} \quad (1)$$

[13] See footnote 8.

$$Fe_3C \text{ (in solution)} + CO_2(gas) \rightleftarrows 3Fe(liq.) + 2CO(gas);$$

$$K_4 = \frac{N^3_{Fe}}{N_{Fe_3C}} \cdot \frac{p^2_{CO}}{p_{CO_2}} \quad (4)$$

$$Fe_3C \text{ (in solution)} \rightleftarrows Fe(liq.) + C \text{ (in solution)}; K_6 = \frac{N^3_{Fe}}{N_{Fe_3C}} \cdot N_C \quad (6)$$

The computed values of K_1, K_4 and K_6 at 1580° C. are 27.0, 7.9×10^3 and 0.038 respectively. The values K_1 and K_4 are reproducible at one atmosphere pressure and 1580° C, and can be obtained by approaching equilibrium from high and low carbon contents and from high and low oxygen contents for several compositions of carbon oxides.

Data on the system liquid iron-hydrogen-water vapor have been used to compute the equilibrium constant of the following equation

$$FeO \text{ (in solution)} + H_2(gas) \rightleftarrows Fe(liq) + H_2O \text{ (gas)};$$

$$K_2 = \frac{N_{Fe}}{N_{FeO}} \cdot \frac{p_{H_2O}}{p_{H_2}} \quad (3)$$

The value of K_2 at 1580° C. is 110. This value is reproducible and can be obtained by approaching equilibrium from high and low oxygen contents.

The accuracy of the values of K_1 and K_2 is established because, when combined as a ratio K_2/K_1, they give a value, 4.07, that is in good agreement with the value, 3.95, for the equilibrium constant of the water-gas reaction. Computations based on the value for the constant K_4 indicate that the carbon content in liquid iron consists of Fe_3C slightly dissociated. This conclusion likewise can be derived from thermodynamic considerations of the iron-carbon diagram. This qualitative agreement with the approved data of the iron-carbon diagram indicates that the value for K_4 is at least of the correct order of magnitude.

The value 0.0025 previously obtained for the carbon-oyxgen product, in liquid iron, at 1600° C. and in contact with mixed carbon oxides at one atmosphere pressure, has been confirmed. Additional experiments have permitted the correlation of the carbon oxides data with the data for the familiar water gas and producer gas reactions. The accuracy of the carbon oxides data, and of the corresponding carbon-oxygen product, 0.0025, is therefore established. This value can be used without appreciable error for carbon and oxygen contents within the limits of 0.01 to 0.5 percent carbon and 0.005 to 0.3 percent oxygen.

WASHINGTON, July 29, 1933.

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U.S. DEPARTMENT OF COMMERCE, BUREAU OF STANDARDS

RESEARCH PAPER RP607

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

A SIMPLE CALORIMETER FOR HEATS OF FUSION. DATA ON THE FUSION OF PSEUDOCUMENE, MESITYLENE (α AND β), HEMIMELLITENE, *o*- AND *m*-XYLENE, AND ON TWO TRANSITIONS OF HEMIMELLITENE

By Frederick D. Rossini

ABSTRACT

A vacuum flask with a thermoelement serves as a simple calorimeter for measuring heats of fusion quickly and economically, with an accuracy of a few percent. The following heats of fusion (with estimated uncertainties), in k-cal. per mole, were obtained: pseudocumene, $-44.1°$ C., 2.75 ± 0.06; hemimellitene, $-25.5°$ C., 2.00 ± 0.05; mesitylene (α), $-44.8°$ C., 2.28 ± 0.06; mesitylene (β), $-51.7°$ C., 1.91 ± 0.05; *o*-xylene, $-25.3°$ C., 3.33 ± 0.07; *m*-xylene, $-47.9°$ C., 2.76 ± 0.05. Hemimellitene was found to have two transitions below the freezing point, with the following heats of transition, in *k*-cal. per mole: hemimellitene ($\gamma \rightarrow \beta$), $-58 \pm 2°$ C., 0.28 ± 0.04; hemimellitene ($\beta \rightarrow \alpha$), $-46 \pm 1°$ C., 0.36 ± 0.04.

CONTENTS

		Page
I.	Introduction	553
II.	Apparatus and method	553
III.	Materials	554
IV.	Standardization experiments	555
V.	Experimental data	557
VI.	Conclusion	559

I. INTRODUCTION

The simple calorimeter described here was assembled in order to provide a means for measuring as quickly and economically as practicable, and with an accuracy of a few percent, the heats of fusion of certain hydrocarbons for which there are no data. Values for the heats of fusion of these substances are required in order to compute the purity of various samples of them from their freezing curves.

II. APPARATUS AND METHOD

The apparatus is shown in figure 1. *A* is an unsilvered vacuum jacketed, pyrex glass flask, the bulb of which has a capacity of about 30 cm^3; *B* is a 2-junction copper-constantan thermoelement sealed with deKhotinsky wax through the rubber stopper *C*; *D* is a cork support.

The principle of the method is to determine the time required to melt a given mass of the substance contained in the flask, when the latter is surrounded by dry air whose temperature is nearly that of the room. The temperature of the air bath is Δt degrees above that of the melting substance. The time rate of flow of heat energy into the flask for any given temperature head, Δt, is determined from experiments with "standard" substances whose heats of fusion are known.

The experimental procedure was as follows: The empty vacuum flask with solid rubber stopper was weighed; the bulb of the flask was filled with the substance under investigation; the flask, with stopper and contents, was weighed; the solid stopper was replaced by one carrying the thermoelement; the bulb of the flask was immersed in liquid air and allowed to remain there until a temperature well below (30 to 60° C.) the freezing point was reached; the flask was then dried and placed in the dry air bath (a glass jar, 12 by 13 by 14 inches, with a close fitting aluminum plate as cover) with the bottom tip of the flask resting on a large cork stopper and the rubber stopper at the top touching a rubber covered lateral support; the emf of the thermoelement was read to 0.01 millivolt at intervals of 2 to 5 minutes except during the melting period, when the intervals were longer.

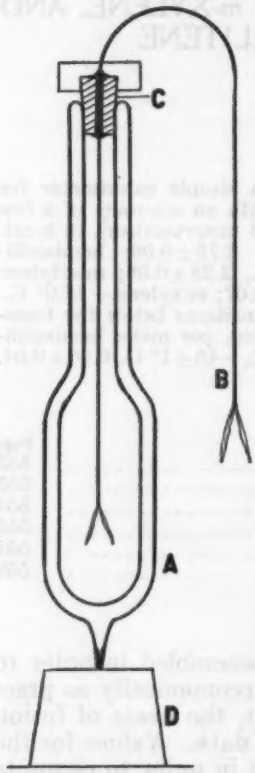

FIGURE 1.—*Sketch of the apparatus*

A, vacuum flask; *B*, 2-junction thermoelement; *C*, rubber stopper; *D*, cork support; length of flask, 8 inches, over all.

The dry air bath was in temperature equilibrium with the air of the laboratory and its temperature stayed about 1° C. below that of the room air. The temperature of the dry air in the bath changed about 0.2 to 0.8° C. during the course of an experiment.

Figure 2 shows a plot of log Δt, where Δt is the difference in temperature between the substance in the flask and the surrounding air, against the time for experiments 23 and 24, with *n*-decane and *n*-octane, respectively. The data required from these plots are (1) the average ordinate during the melting period, the antilogarithm of which is Δt_l, and (2) the length of the abscissa (through this average ordinate) intercepted by the two straight lines, which is z_l, the time of melting.

If m is the mass of the substance in the flask, and l_l is its heat of fusion in cal. per g, then, at the given temperature head of Δt_l, the rate of flow of heat energy into the flask from its surrounding air, during the melting period, is

$$A = ml_l/z_l \text{ cal. min.}^{-1}$$

Having determined the value of A for various values of Δt_l from experiments with "standard" substances, then for the "unknown" substance

$$l_l = z_l A/m \qquad \text{cal. g}^{-1}$$

III. MATERIALS

The hydrocarbons employed in the present investigation were as follows: *n*-octane (F.P. −56.8° C.) and *n*-dodecane (F.P. −9.8° C.) prepared synthetically by Mair [1]; *n*-decane (F.P. −29.8° C.) pre-

[1] Mair, B. J., B.S.Jour. Research, vol. 9, p. 457, 1932.

pared from petroleum by Bruun and Hicks-Bruun [2]; benzene prepared by the Bureau des Étalons Physico-Chimiques, University of Brussels; *o*-xylene (F.P. −25.3° C.) and *m*-xylene (F.P. −47.9° C.) prepared from petroleum by White and Rose [3]; pseudocumene (1, 2, 4-trimethylbenzene, F.P. −44.1°C.), mesitylene (1, 3, 5-trimethylbenzene, F.P. −44.8° C.), and hemimellitene (1, 2, 3-trimethyl-

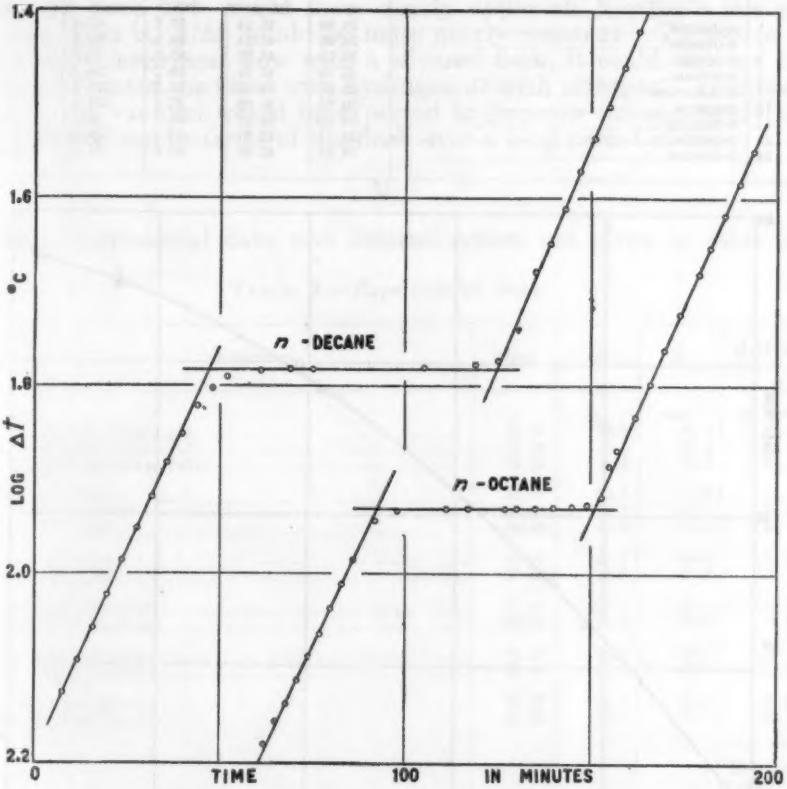

FIGURE 2.—*Curves for n-octane and n-decane.*
The scale of ordinates gives log Δt in °C. The scale of abscissae gives the time in minutes.

benzene, F.P. −25.5° C.) prepared from petroleum by Mair and Schicktanz [4].

IV. STANDARDIZATION EXPERIMENTS

Table 1 gives the data obtained in the experiments with the "standard" substances, values for whose heats of fusion were taken from the data of Parks and coworkers.[5]

[2] Bruun, J. H., and Hicks-Bruun, M. M., B.S.Jour. Research, vol. 8, p. 583, 1932.
[3] White, J. D., and Rose, F. W., Jr., B.S.Jour. Research, vol. 9, p. 711, 1932.
[4] Mair, B. J., and Schicktanz, S. T., B.S.Jour. Research, vol. 11, 1933.
[5] (*a*) Huffmann, H. M., Parks, G. S., and Barmore, M., J.Am.Chem.Soc., vol. 53, p. 3876, 1931; (*b*) Huffmann, H. M., Parks, G. S., and Daniels, A. C., J.Am.Chem.Soc., vol. 52, p. 1547, 1930.

TABLE 1.—*Data from the Standardizing Experiments*

Experiment no.	Substance	Mass	Value for the heat of fusion (from Parks et al.)	Δt_f	z_f	A
		g	*cal. g*$^{-1}$	°*C.*	*min.*	*cal. min.*$^{-1}$
1	n-decane	24.99	48.34	57.7	85.4	14.14
2	n-decane	24.99	48.34	58.1	85.3	14.16
3	n-octane	23.19	43.21	85.7	57.0	17.58
4	n-octane	23.19	43.21	85.7	56.2	17.83
11	n-dodecane	25.57	51.33	36.3	129.4	10.14
12	benzene	29.53	30.09	22.5	138.5	6.42
13	n-nonane	24.80	41.21	85.7	56.5	18.09
23	n-octane	24.19	43.21	85.1	58.9	17.74
24	n-decane	24.13	48.34	61.0	77.9	14.97

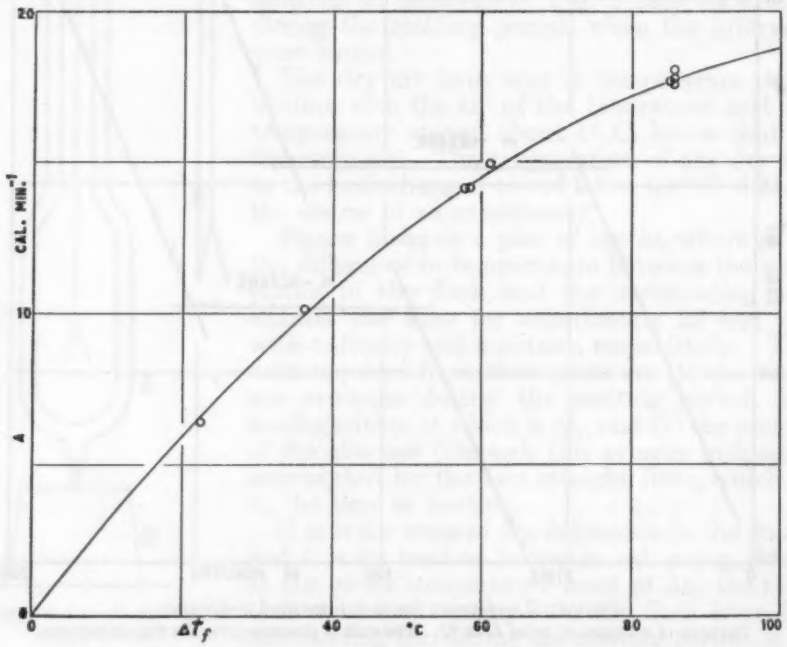

FIGURE 3.—*Plot of the data from the standardizing experiments.*

The scale of ordinates gives A, the heat flow into the flask, in cal. per min. The scale of abscissae gives Δt_f, the difference between the temperature of the substance at its melting point and that of the air surrounding the flask, in °C.

The experiments with the "standard" substances were run both before and after the experiments with the "unknowns", and the thermal conductance of the flask was apparently unchanged during the course of the experiments, which extended over a period of 4 weeks.

The data from the standardizing experiments are plotted in figure 3, where A, the heat flow in cal. per minute, is plotted againsy Δt_f, the difference in temperature between the substance at its melting point and the air surrounding the flask. The equation of the curve is given by

$$A = 0.323\ \Delta t_f - 0.00135\ (\Delta t_f)^2$$

The flask used in the present investigation served very well for substances freezing in the range 0° to −70° C. For lower temperatures a flask with a smaller heat leak would be better. For substances melting above room temperature, the reverse process of starting the experiment with the flask containing the liquid substance at a temperature of 30° to 60° above the melting point and letting it cool down to the room temperature should be feasible. With a silvered flask the heat flow would more nearly approach Newton's law of cooling, that is, $A/\Delta t_f$ would be more nearly constant. To secure a sufficiently large heat flow with a silvered flask, it could, instead of being evacuated, be filled with hydrogen or with nitrogen. Elimination of the vacuum would be expected to improve the constancy of the thermal conductance of the flask over a long period of time.

V.

The experimental data and determinations are given in table 2.

TABLE 2.—*Experimental Data*

Exp. no.	Substance	Mass	Δt_f	z_f	Heat of fusion
		g	*°C.*	*min.*	*cal. g⁻¹*
5	Hemimellitene (α)	29.70	56.6	34.9	16.40
6	Hemimellitene (α)	29.70	53.5	38.3	17.30
7	Hemimellitene (α)	29.70	49.8	38.6	16.56
10	*o*-Xylene	30.70	50.1	75.3	31.39
22	*o*-Xylene	27.02	52.5	65.5	32.09
25	*o*-Xylene	30.43	53.0	70.3	30.77
14	*m*-Xylene	29.92	75.3	46.3	25.78
15	*m*-Xylene	29.92	75.3	47.0	26.17
16	Mesitylene (β)	29.50	77.1	27.6	15.79
17	Mesitylene (β)	29.50	77.4	28.0	16.05
18	Pseudocumene	28.62	72.4	40.7	23.19
19	Pseudocumene	28.62	74.8	39.1	22.69
20	Mesitylene (α)	28.75	72.1	33.4	18.90
21	Mesitylene (α)	28.38	72.8	33.7	19.42
					Heat of transition
7	Hemimellitene ($\gamma \rightarrow \beta$)	29.70	78.9	4.0	2.30
7	Hemimellitene ($\beta \rightarrow \alpha$)	29.70	66.5	5.8	3.03

In figure 4 are plotted the data from experiments 7 and 15, with hemimellitene and *m*-xylene, respectively. As indicated by the curve in figure 4, hemimellitene has three stable solid forms. Values for the two heats of transition were computed from this curve. From these and other experiments the transition temperatures for hemimellitene were determined to be -46 ± 1 and $-58 \pm 2°$ C. In the case of mesitylene, Mair and Schicktanz [6] found both a stable (F.P. −44.8° C.) and a metastable crystalline form (F.P. −51.7° C.). The heats of fusion of both of these crystalline forms of mesitylene were determined.

[6] See footnote 4, p. 555.

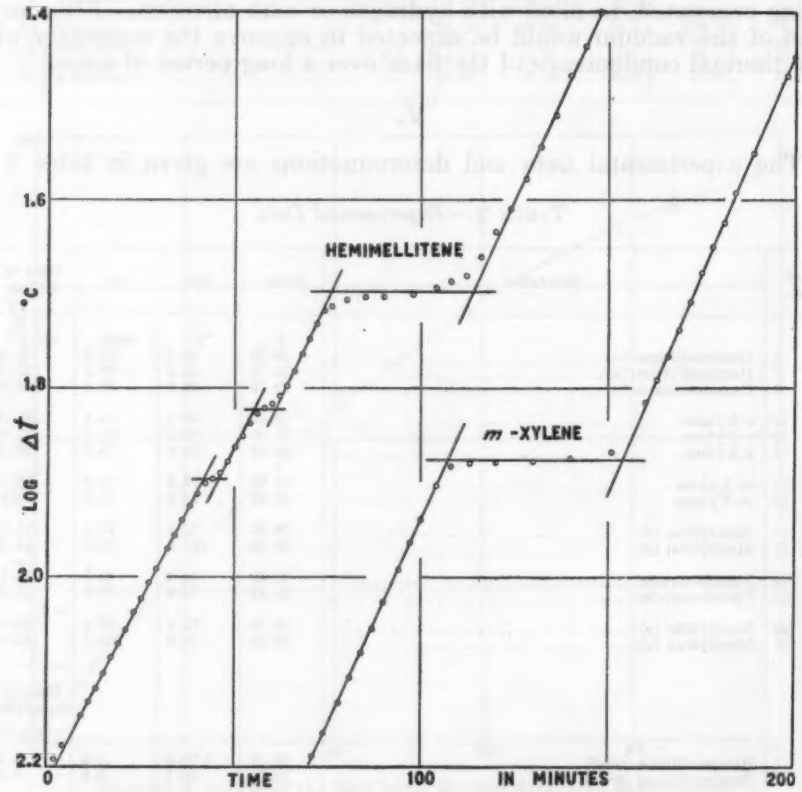

FIGURE 4.—*Curves for hemimellitene and m-xylene.*

The scale of ordinates gives log Δt in °C. The scale of abscissas gives the time in minutes.

TABLE 3.—*Heats of Fusion*

Substance	Molecular weight	F.P.	Heat of fusion (with estimated uncertainty)
	g	° *C.*	*k-cal. mole*$^{-1}$
Pseudocumene	120.09	−44.1	2.75±0.06
Hemimellitene (α)	120.09	−25.5	2.00±0.05
Mesitylene (α)	120.09	−44.8	2.28±0.06
Mesitylene (β)	120.09	−51.7	1.91±0.05
o-Xylene	106.08	−25.3	3.33±0.07
m-Xylene	106.08	−47.9	2.76±0.05

TABLE 4.—*Heats of Transition*

Substance	Transition temperature	Heat of transition (with estimated uncertainty)
	° *C.*	*k-cal. mole*$^{-1}$
Hemimellitene (γ→β)	−58±2	0.28±0.04
Hemimellitene (β→α)	−46±1	0.36±0.04

VI. CONCLUSION

The values obtained from the present investigation, together with the estimated uncertainties, are given in tables 3 and 4. No other data exist for hemimellitene and mesitylene. Parks and coworkers [7] found for *m*-xylene [8] (F.P. −53.5° C.) 2.73, for *o*-xylene (F.P. −25.3° C.) 3.11, for pseudocumene (F.P. −44.5° C.) 3.02, k-cal. per mole.

WASHINGTON, July 28, 1933.

[7] See footnote 5, p. 555.

[8] Huffman, Parks, and Daniels (see footnotes 5b, p. 555) state that their *m*-xylene was not very pure. Their sample froze 5.6° C. below the freezing point of the sample used in the present investigation.

U.S. DEPARTMENT OF COMMERCE, BUREAU OF STANDARDS

RESEARCH PAPER RP608

Part of Bureau of Standards Journal of Research, Vol. 11, October 1933

NOTE ON A MULTI-FREQUENCY AUTOMATIC RECORDER OF IONOSPHERE HEIGHTS[1]

By T. R. Gilliland

ABSTRACT

A system is described which gives a curve of virtual height of the ionosphere against frequency. The pulse method of Breit and Tuve is employed with modifications. Short pulses of radio-frequency energy are transmitted and the time required for the energy to go up and return is recorded automatically by a galvanometer oscillograph of the type previously used for fixed-frequency work. The transmitting and receiving sets are shifted in frequency from 2,500 to 4,400 kc/s at the uniform rate of 200 kc/s per minute.

Records are presented which show the characteristics for different times of day and night. In the daytime, during the period of these tests, three strata were usually indicated. For the lower range of frequencies, reflections come from the E layer with a virtual height of around 120 km. As frequency is increased the waves pass through the E layer and are returned from the F_1 layer with virtual heights of the order of 200 km. The frequency for which this transition takes place varies with time of day and with season. In the middle of the day, during these tests, this critical frequency was in the neighborhood of 3,000 kc/s, while the critical frequency for passing through the F_1 to the F_2 layer was usually between 3,800 and 4,100 kc/s. The F_2 layer shows virtual heights of 280 km or more. Of particular interest is the character of the change observed when passing from one stratum to another as the frequency is increased. Although, at times, when passing from E to F_1 reflections may drop out completely for a short interval, frequently the curve is continuous and the time retardation will reach a high value just before the appearance of the F_1 reflection. When passing from F_1 to F_2 the virtual height frequently reaches 800 or 900 km. As evening approaches reflections no longer come from the E layer for the frequencies 2,500 kc/s to 4,400 kc/s, and the long retardation between F_1 and F_2 becomes less pronounced. By sunset the curve is almost straight and there is little change of height with frequency. Later at night the highest frequencies cease to be returned and long retardations again occur. The phenomenon of double refraction is in evidence at this time.

The system described here offers a more convenient method than the manual methods previously employed and greater economy is obtained in both time and personnel. It is possible to obtain records with much greater detail.

The results are presented with the idea of indicating the value of this method in the study of the physical properties of the upper atmosphere as well as in the study of radio transmission.

CONTENTS

		Page
I.	Introduction	562
II.	Description of multi-frequency system	563
III.	Results	565
IV.	Conclusion	566

[1] Presented in part at meeting of American Section, International Scientific Radio Union, at Washington, April 27, 1933, and also presented at Eighth Annual Convention, Institute of Radio Engineers, at Chicago, June 27, 1933.

I. INTRODUCTION

The purpose of this note is to describe a system for automatically recording the virtual heights of the ionized layers of the upper atmosphere for a given band of radio frequencies and to show the type of record obtained. The system utilizes the pulse method of Breit and Tuve [2] with modifications which permit automatic recording and the shifting of the transmitting and receiving sets continuously over a desired band of frequencies.

Observers in previous work have recorded virtual heights manually where information was desired for more than one frequency. The frequency was varied by steps, and readings of virtual height were made for each step. The disadvantages with this method are that it requires considerable time and labor and, since changes in the ionosphere are often quite rapid, important details are likely to be missed. Critical effects are often observed for very small changes in frequency, so that a system that will give a continuous and rapid frequency variation is desirable.

The importance of the ionosphere in radio transmission has led to an intensive study of its characteristics both in this country and abroad. An investigation which gives fundamental information about its physical properties is of value in the general study of the atmosphere as well as in its application to radio transmission. The system originated by Breit and Tuve for making these studies consists of a radio transmitter, receiving set, and galvanometer oscillograph with photographic attachment. The transmitter is made to send out short pulses, which arrive at the receiving set via the ionosphere as well as by a path along the ground. By passing the output of the receiving set through the oscillograph, a photographic record is made which gives a measure of the time required for the pulse to go up and back. This time interval is used to calculate the "virtual" height.

This method has been modified for automatic recording at a fixed frequency.[3] [4] This is done by keying the pulses at the transmitter with a chopper driven by a synchronous motor. At the receiving station the oscillograph is provided with a revolving mirror also driven by a synchronous motor connected to the same power system. With this arrangement the pulse pattern is projected onto the oscillograph screen so that the ground-wave pulse remains stationary while the sky-wave pulses shift with respect to it as the virtual height of the ionosphere changes. In order to make a record of the changes taking place, photographic paper is passed over the pulse pattern in a direction parallel to the axis of the revolving mirror. The pattern is masked so that the paper is exposed to only the top portion of the pulses. The usual cylindrical lens of the oscillograph optical system is replaced by a spherical lens so that the light from the top part of the pulse is focused into a small spot on the paper. As the paper moves the ground pulse will trace a straight line since its path length is fixed. As the ionosphere shifts in virtual height, the time required for the other pulses to return will change and traces will be made which vary in distance from the ground trace. This distance gives a direct measure of the virtual height. In the present arrangement

[2] Breit and Tuve, Phys. Rev., vol. 21, p. 554, 1926. Proc. I.R.E., vol. 16, p. 1236, 1928.
[3] Gilliland and Kenrick, B.S.Jour.Research, vol. 7, p. 783, Nov. 1931; Proc.I.R.E., vol. 20, p. 540, March 1932.
[4] Gilliland, B.S.Jour.Research, vol. 11, p. 141, July 1933.

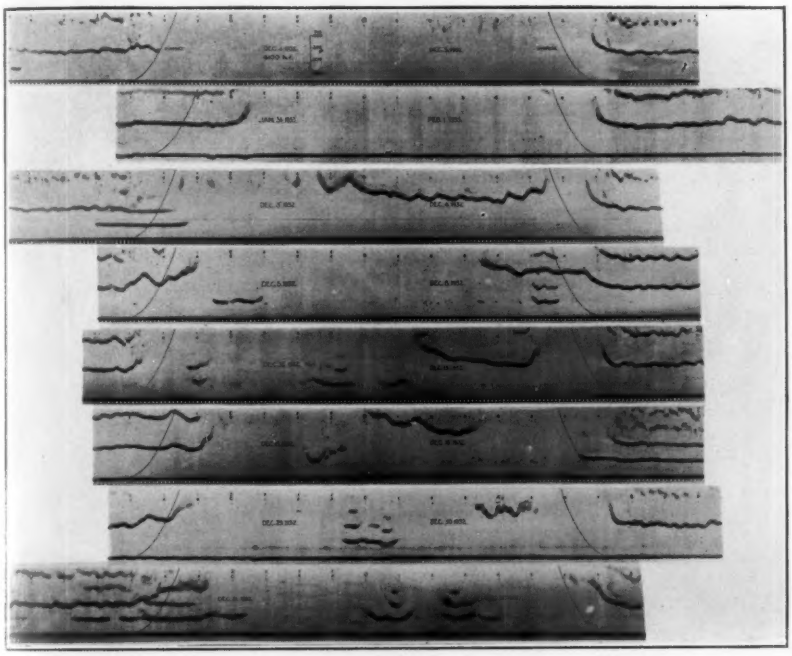

FIGURE 1.—*Photograph showing type of record obtained on a fixed frequency of 4,100 kc.*

The first two records show almost no reflections at night. Third to seventh records, inclusive, show night reflections from both the E and the F layers. The last record shows reflections from only the E layer at night.

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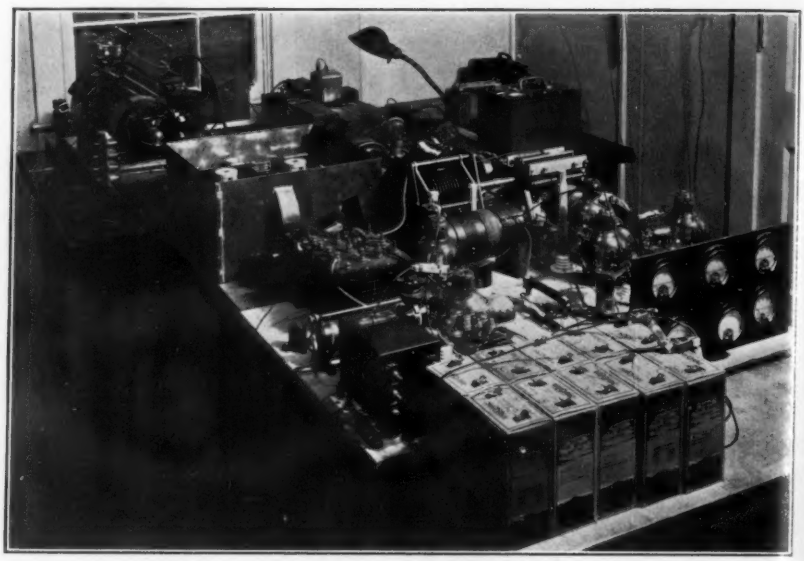

FIGURE 4.—*Photograph showing preliminary arrangement.*

Transmitter in foreground, receiving set converter in box with cover removed, recorder in background at left.

the transmitting and receiving sets are placed in the same room and the chopper wheel and revolving mirror are attached to the same shaft. The placing of the complete equipment in one room adds greatly to the simplicity of operation, especially with the multi-frequency system. Troubles from phase shifts in the power system are eliminated.

Figure 1 shows the type of record obtained on eight different days for a fixed frequency of 4,100 kc/s. Here reflections are shown from both the *E* and the *F* layers. During the daytime on these days reflections came more often from the *F* layer showing virtual heights of 240 km and higher. Reflections from the *E* region at about 100 to 130 km were found to occur at irregular intervals.[5]

II. DESCRIPTION OF MULTI-FREQUENCY SYSTEM

The recorder for the multi-frequency system is the same as that used for fixed-frequency work. The receiving set is also the same except that a mechanism is introduced for tuning over the desired range of frequencies. A superheterodyne circuit is employed with a broadcast receiving set and high-frequency converter. The galvanometer oscillograph is connected in series with the moving coil of the

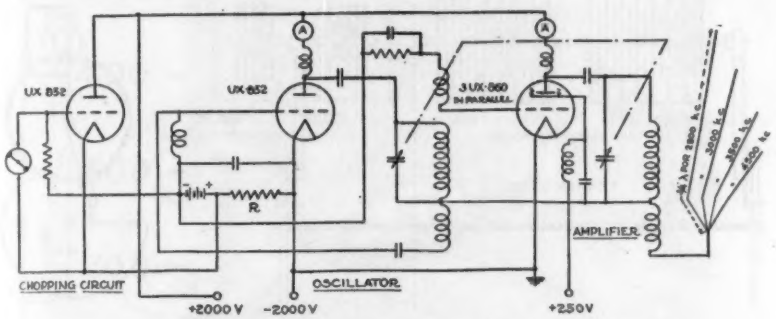

FIGURE 2.—*Circuit arrangement of transmitter.*

With the chopper contacts open, the oscillator grid is biased below the oscillation condition by the *C* battery. When the contacts are closed, current flows in the plate circuit of the chopping tube which includes resistance *R*. Current through *R* brings the oscillator grid up to the oscillation point and at the same time causes the bias on the amplifier grids to assume the proper operating value.

loudspeaker. The circuit arrangement of the transmitter is shown in figure 2. The oscillator circuit using one type UX-852 tube is followed by a single amplifier stage using three type UX-860 tubes in parallel. One UX-852 is employed as a chopping tube to avoid sparking at the chopper contacts which occurs when the oscillator is chopped directly. With the chopper contacts open, the oscillator grid is biased below the oscillation condition by the *C* battery. When the contacts are closed current flows in the plate circuit of the chopping tube which includes resistance *R*. The current through *R* brings the oscillator grid up to the oscillation point and at the same time causes the bias on the amplifier grids to assume the proper operating value. The chopper is geared so that about 10 pulses per second are transmitted. The rotors of the oscillator and amplifier tuning condensers are attached to the same shaft.

The antenna system of the transmitter consisted of inverted *L* antennas of three-fourths wave length for 2,500, 3,000, 3,500, and

[5] See footnote 4.

4,500 kc/s, all connected permanently together at the base. The receiving antenna was a single inverted L of three-fourths wave length for about 4,000 kc/s.

The mechanical arrangement is shown schematically in figure 3. The transmitter tuning condensers are actuated by one cam while the receiving set condensers are actuated by another. Both cams are attached to a single shaft driven by a synchronous motor through a reduction gear. The cam lever of the transmitter is geared to the condenser shaft by spur gears with a step-up ratio of 4 to 1 so that the rotors turn through 180° while the cam lever moves through 45°. The rotors of the oscillator and detector tuning condensers of the receiving set are also attached to a single shaft. For the desired range of frequencies these rotors turn through more than 180° so that a slightly higher gear ratio is used.

It was found that one set of coils of the receiving set could be worked conveniently between 2,500 kc/s and 4,400 kc/s without modification and since this is an interesting range for daytime work, the transmitter was designed to cover this range. The cam shapes were determined after the mechanism had been assembled. The trans-

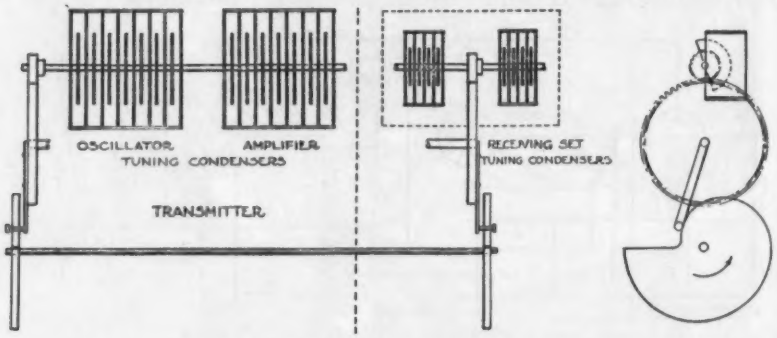

FIGURE 3.—*Schematic diagram of mechanical arrangement.*

Transmitter condensers are actuated by one cam and receiving-set condensers are actuaed by another. Both cams are attached to a single shaft and are driven by a synchronous motor through a reduction gear.

mitter cam shape was determined by changing the transmitter frequency in small steps. The distance from the cam roll to the center of the cam shaft was measured for each step with a caliper. Having chosen 200 kc per minute as the rate of change of frequency, the angular position of the cam was known for each small step. With the radius known for each angular position it was then possible to lay out this cam. In order to lay out the other cam, the receiving set was tuned to the transmitter as the frequency was varied in small steps and the distance from the cam roll to the center of the cam shaft was measured for each step. With these measurements and the known rate of rotation, this cam could be laid out. The cam was cut slightly large and then filed down to size after being installed. The tuning of the receiving set to transmitter frequency for each small step can be done conveniently while the transmitter is being pulsed. It was found advisable to keep the receiving set at low gain while tuning so that only the direct pulse was visible on the oscillograph screen. If tuning is done at high gain with reference to the sky wave pulses, inaccurate settings may be obtained at critical frequencies.

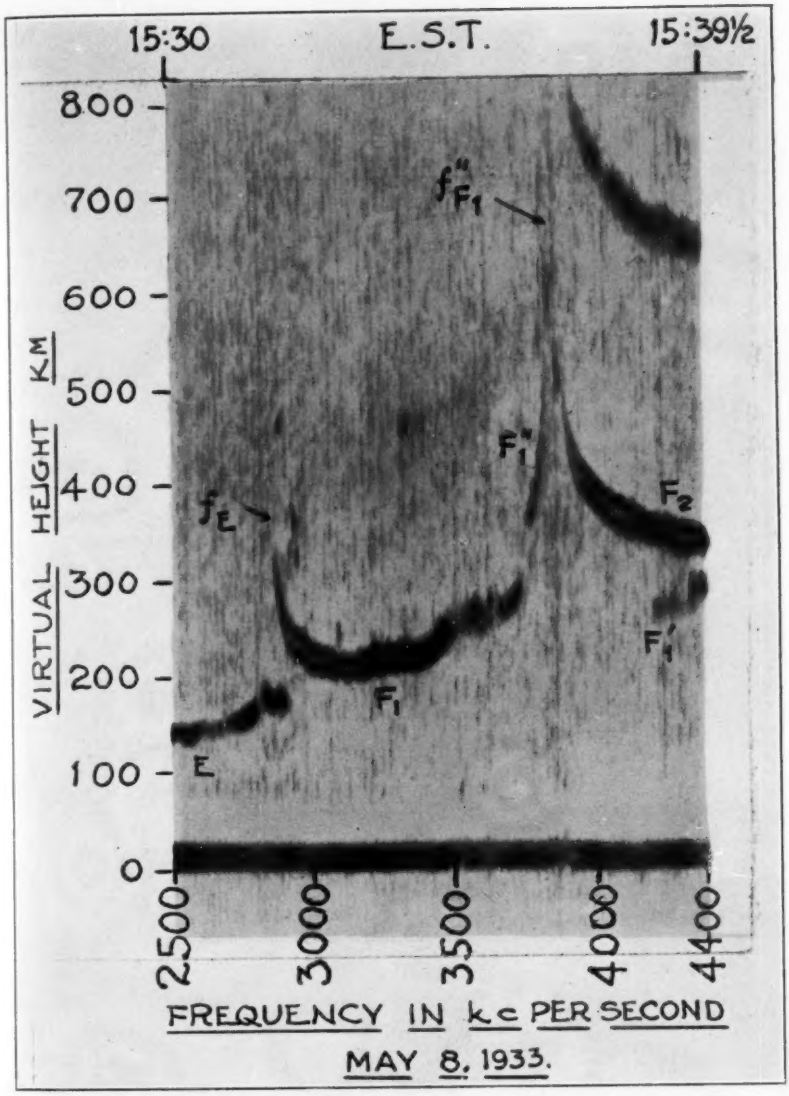

FIGURE 5.—*Photograph showing type of record obtained in daytime.*

f_E denotes critical frequency for E layer. f'_{F1} denotes critical frequency for ordinary ray in F_1 layer.

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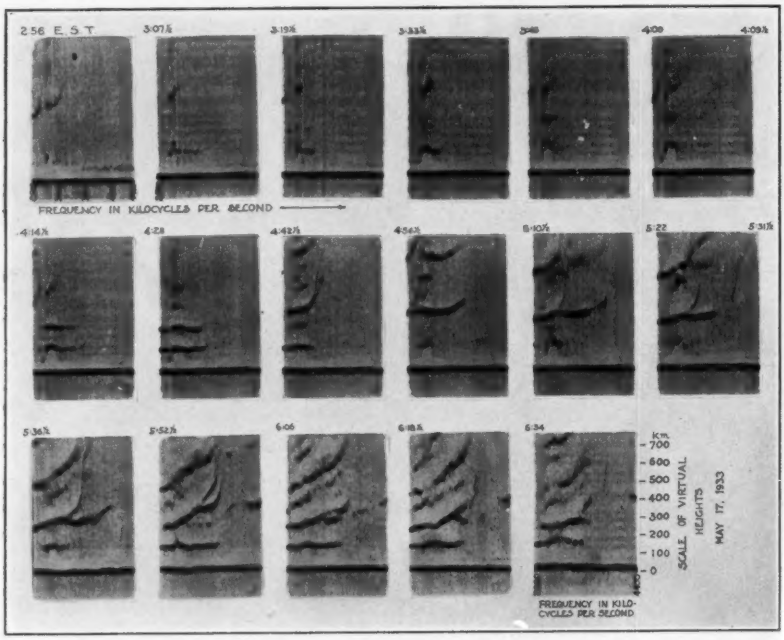

FIGURE 6.—*Records of May 17.*

Each short section represents a frequency band from 2,500 to 4,400 kc and requires 9½ minutes. Last two records in second row show critical effects for ordinary and extraordinary rays.

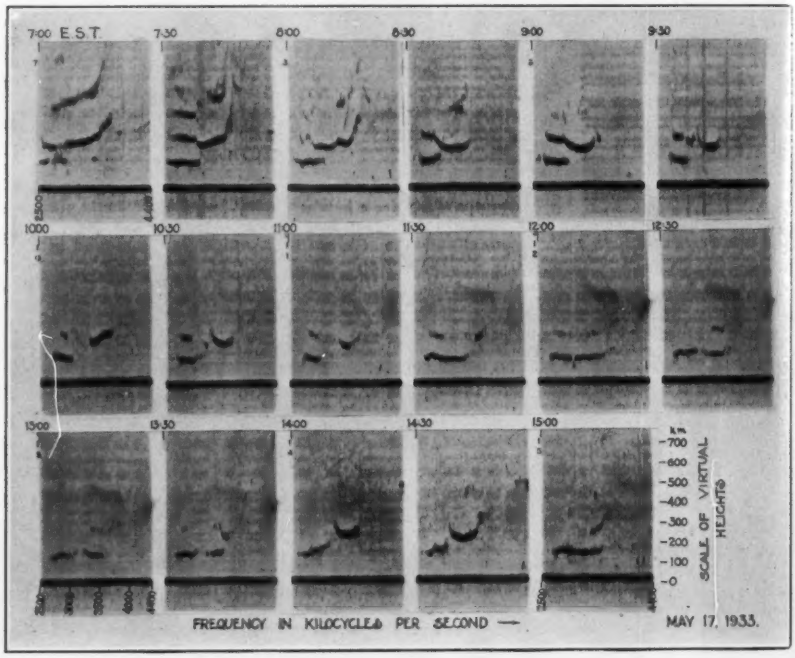

FIGURE 7.—*Records for May 17 (continued).*

F_1 reflections were very weak during this day.

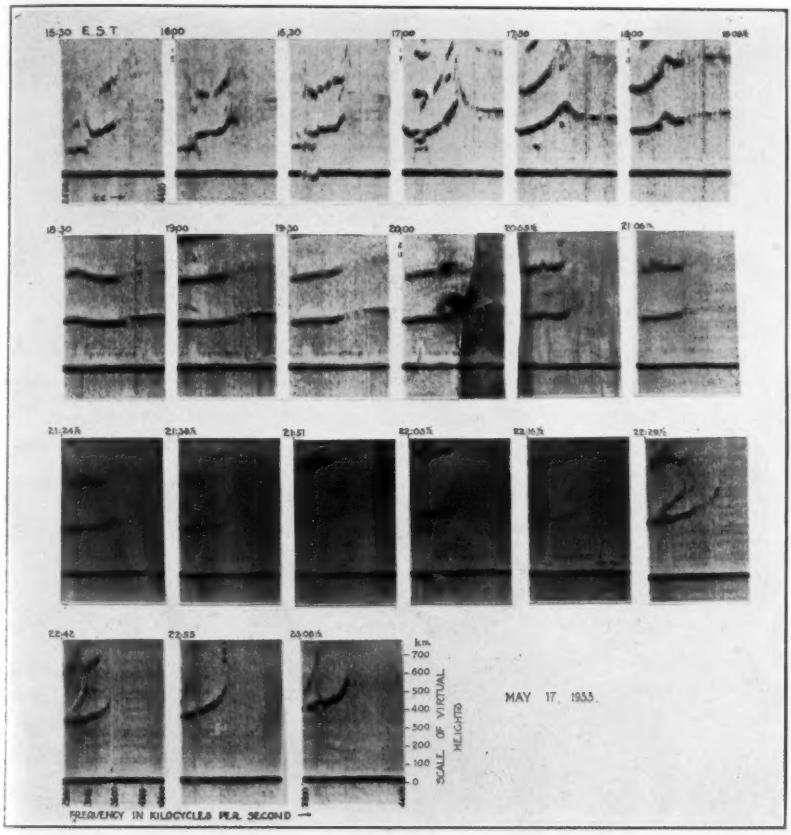

FIGURE 8.—*Records for May 17 (continued).*

Note appearance of F_1 stratification at 1700 which disappears later in evening. The ordinary and extraordinary rays become critical in this band again before midnight.

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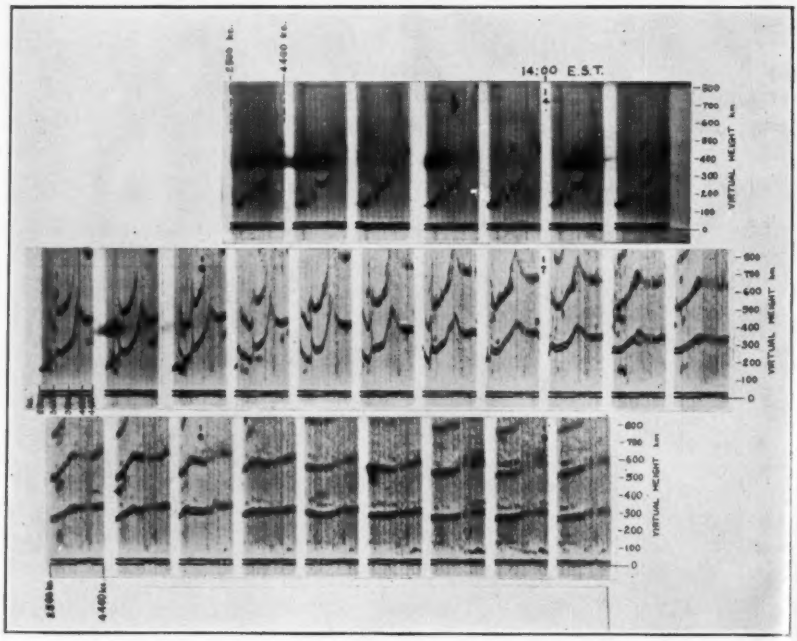

FIGURE 9.—*Photograph of records taken during afternoon and evening of April 22.*
Note double critical effect between E and F_1 layer in first row.

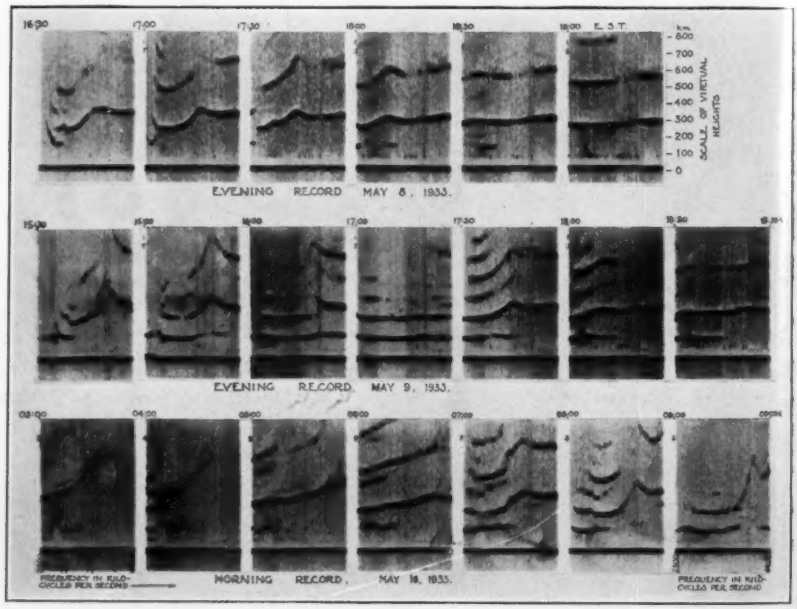
E. S. T.
SCALE OF VIRTUAL HEIGHTS
EVENING RECORD MAY 8, 1933.
EVENING RECORD MAY 9, 1933.
MORNING RECORD, MAY 16, 1933.
FREQUENCY IN KILOCYCLES PER SECOND

FIGURE 10.—*Records showing changes during two evening runs and one morning run.*
Records for morning of May 16 show beginning of F_1 stratification.

The system is adapted for sweeping automatically through the band either once each hour or each half hour. If switching is done manually at the end of each run, it is possible to sweep through the band almost six times per hour.

Figure 4 is a photograph of the preliminary arrangement of the transmitter, receiving set, and recorder.

III. RESULTS

The type of record obtained in the daytime is shown in figure 5. Here the notation followed is that adopted by Kirby, Berkner, and Stuart in a paper now in preparation. The single and double prime letters indicate extraordinary and ordinary rays, respectively, in accordance with the notation of Appleton and Builder.[6] Figure 5 shows the changes in virtual height as the frenquency is changed uniformly from 2,500 to 4,400 kc/s at the rate of 200 kc/s per minute. E layer reflections are noted at the left coming from a virtual height of about 135 km for 2,500 kc/s. The virtual height increases gradually as the frequency is increased to about 2,850 kc/s. This is the critical frequency f_E for the E layer, and long retardations occur. As frequency is increased above this point, the virtual height drops rapidly to the level of the F_1 layer. At about 3,200 kc/s, the virtual height reaches a minimum of 210 km for the F_1 layer. As the frequency is increased the virtual height of the F_1 layer for the ordinary ray (F''_1) increases until another critical value f''_{F1} is reached at about 3,850 kc/s and the virtual height reaches 700 km. This critical frequency is thought to be that for the ordinary ray as it passes through the F_1 layer. As frequency is increased still farther, reflections for this ray come from the F_2 region showing virtual heights of about 330 km. The trace at the right marked F_1' is thought to be the extraordinary ray for the F_1 layer. It is usually relatively weak and frequently does not appear at all. From observations on higher frequencies, Kirby, Berkner, and Stuart have noted that this ray becomes critical at a frequency approximately 800 kc/s higher than for the ordinary ray during the daytime. It is likely that the critical frequency f_E noted for the E layer is for the ordinary ray and that the extraordinary ray is absorbed. The trace in the upper right hand corner is a multiple of the F_2 trace indicating that the pulse energy has gone up to the F_2 layer and back twice. As evening approaches the stratification indicated in the F region disappears and it appears to become a single layer.

Figures 6, 7, and 8 show records taken over a period of about 21 hours. Each short section of record represents the frequency band of 2,500 to 4,400 kc/s and requires 9½ minutes. In figure 6 weak reflections are recorded for both the E and the F regions until about 0415 when strong E reflections appear. By 0456, the E reflections have practically disappeared and strong F reflections are recorded. The records beginning at 0510½ and 0522 show the critical effects for the two rays. It has been suggested that the frequency separation between these two rays, when critical, should give a measure of the strength of the earth's magnetic field at the layer.[7] Results during the remainder of the morning and the early afternoon should not be taken as typical. Apparently abnormally strong E layer ionization

[6] Appleton and Builder. Proc. Phys. Soc., vol. 45, part 2, p. 208, 1933.
[7] A theoretical discussion of this point is now in preparation by Mr. K. A. Norton.

was occurring at intervals and F layer reflections were very weak at the higher frequencies. The absence of F_1 reflections during the middle of the day was checked by manual observations with other equipment. By 1700 (fig. 8) the E critical frequency is just below 2,500 kc/s and F region stratification is now in evidence. The stratification gradually becomes less pronounced and has practically disappeared by 1830, which is 48 minutes before sunset at the ground. Critical effects for the two rays appear before midnight. The failure to record reflections between 3,500 and 4,000 kc/s at times is caused by interference from the large number of amateur stations in this band.

Figure 9 shows records taken during the afternoon and evening of April 22. The records in the first row show a double critical effect between the usual E and F_1 layers, indicating another stratum, making a total of four strata at this particular time. The changes indicated at the left in the second row are complex and very sharp critical effects are noted. As evening approaches the E layer critical frequency goes below 2,500 kc/s and the F region stratification gradually disappears.

Figure 10 shows records taken during two evenings and one morning. The records for the evening of May 9 show the appearance of strong E reflections which obscure F reflections for a time. The morning records for May 16 show the beginning of the F region stratification.

IV. CONCLUSION

In this note, preliminary results have been presented with the view to indicating the possibilities offered by this method in the study of radio transmission and physical properties of the upper atmosphere. It is believed that records of the type shown give a more complete picture than it has been possible to obtain by methods previously used. It is hoped that it will be possible to continue these observations and to cover a wider band of frequencies.

WASHINGTON, June 17, 1933.

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